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SPACE SHUTTLE SEAL MATERIAL AND DESIGN DEVELOPMENT FOR EARTH STORABLE PROPELLANT SYSTEMS

FINAL REPORT

OCTOBER 1973

Prepared for

NATIONAL AERONAUTICS AND SPACE ADMINIS JOHNSON SPACECRAFT CENTER MOUSTON, TEXAS, 77058

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FOREWORD

This program was conducted by TRW Systems Group, Redondo Beach, California, under contract to the NASA Johnson Spacecraft Center, Houston, Texas. The contract number was NAS 9-12729.

The NASA program manager was Mr. J. W. Griffin.

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Personnel at TRW Systems responsible for the conduct of the program include: Mr. J. W. Martin and Mr. J. R. Denson, Applied Chemistry Department; Mr. R. N. Porter, Mr. F. L. Merritt and Mr. H. M. Elmendorf of the Chemical Propulsion Department. Administrative and technical support was provided by Dr. E. A. Burns, Manager, Applied Chemistry Department; Mr. H. Macklis, Assistant Manager, Chemical Propulsion Department; and Mr. D. F. Reeves, Head, Propulsion Auxiliaries Section. The program manager was Mr. R. G. Gilroy.

ABSTRACT

This report provides the results of a program to investigate and characterize seal materials suitable for Space Shuttle Storable Propellant Systems.

Two new elastomeric materials were identified as being potentially superior to existing state-of-the art materials for specific sealing applications. These materials were Af-E-124D and AF-E-411. AF-E-124D is a cured perfluorinated polymer suitable for use with dinitrogen tetroxide oxidizer, and hydrazine base fuels. AF-E-411 is an ethylene propylene terpolymer material for hydrazine base fuel service.

Data are presented relative to low and high temperature characteritics as well as propellant exposure effects. Types of data included are: mechanical properties, stress strain curves, friction and wear characteristics, compression set and permeability. Sealing tests with a flat poppet-seal valve were conducted for verification of sealing capability.

A Seal Design Procedure is provided which contains a step-by-step procedure for designing seals. The information needed for seal design is primarily summarized and presented as well as references for more in-depth information.

A plan for a controlled method of investigating contamination sensitivity is also presented.

Preliminary Specifications for AF-E-124D and AF-E-411 are also provided. Limited properties data are also provided for the following state-of-the art seal materials: Teflon, Kel-F, Kynar, Vespel, Ethylene Propylene Rubber, Carboxynitroso Rubber and Viton.

The bibliography includes over 200 references relating to seal design or materials and presents a concise tabulation of the more useful seal design data sources.

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1.0 INTRODUCTION

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Seals for Space Shuttle storable propellant applications have the unique problem of extended life durations over those required for previous applications such as Apollo. This requires the capability of extended exposure to propellants and a higher number of operating cooles. The minimum shelf life (installed) is anticipated to be seven years, and the service life, five years. In addition, the operational concept does not provide for flushing or decontaminating between missions, although this must be considered a possibility.

Present state-of-the-art materials which could be used each have one or more deficiencies, particularly for oxidizer applications. Through previous programs oriented toward cryogenic seal development (Contracts NAS 9-11866, NAS 9-10481, and NAS 9-12500) new elastomeric materials have been identified which appear promising for storable propellant applications. Some data from those programs, particularly NAS 9-12500, are included in this report. This program was oriented toward further investigation and characterization of these types of materials, and comparison with existing state-of-the-art seal materials.

The objective of this program was to evaluate, investigate and characterize seal materials and designs which would be suitable for long-term use in Space Shuttle earth storable propellant systems. The propellants of interest are nitrogen tetroxide (N_2O_4), hydrazine (N_2H_4), unsymmetrical dimethylhydrazine (UDMH), 50/50 blend of hydrazine and unsymmetrical dimethylhydrazine (N_2H_4 /UDMH), and monomethylhydrazine(MMH).

The range of temperatures investigated during this program were from -100°F to +300°F. Other factors considered were: effects of long-term propellant exposure, effects of cleaning solvents; mechanical property retention and combinations of these factors.

This report presents the results of this program and provides recommendations for further seel material and design development. Included in the report are the results of the state-of-the-art survey of seal materials; the analysis of critical seal parameters; a compilation of the test data obtained in this program and the previous cryogenic seal program under contract NAS 9-12500 and a summary of data relating to other state-of-the-art seal materials not tested under this program.

A seal design procedure guide has been prepared as part of this program, providing a compilation of reference data and techniques for seal design.

The seal design problem is, to a large degree, a function of available materials. A major problem in designing a seal is to accommodate or strengthen basic seal material weaknesses. An improvement in material properties and characteristics can greatly reduce the complexity of a seal design. During the course of this program, emphasis was placed on improving the seal material to a realistic maximum and then characterizing the material to determine its performance limitations.

2. Summary

2.0 SUMMARY

The purpose of this program was to develop and characterize seal materials and to provide design information for Space Shuttle Storable Propellant sealing applications. As a result of previous programs (Refs. 89 and 90), the effort was oriented toward non-metallic seals including improved elastomeric materials. Two elastomeric materials were identified as most promising and were characterized to allow evaluation of capability as compared with the best state-of-the-art material, Teflon. Teflon has proven to be a good general seal material for storable propellant applications, as shown by the many designs for which it has been selected. Typical cases are summarized in this report. The primary advantages of Teflon are its relative inertness, and generally high retention of mechanical properties over a wide range of conditions. Disadvantages include a tendancy to creep under load, a tendency to toward contamination sensitivity (dependent upon design) and a tendency to flake under wear applications.

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The basic advantages of elastomeric materials (as compared with Teflon) are resiliency, and the ability to "recover" after being compressed for long periods of time. Another advantage is a tendency toward greater contamination tolerance, although this parameter was not investigated as part of this program.

The program results show that AF-E-124D is a suitable seal material for either nitrogen tetroxide oxidizer or hydrazine base fuel service. Good mechanical and chemical property retention were demonstrated over varying time periods. A limitation of AF-E-124D is reduction of mechanical properties at elevated temperature. Although no chemical change occurs, mechanical property reduction above approximately 200°F must be accommodated in design.

AF-E-411 appears to be an excellent material over the tested temperature range of -100° to +300°F and in contact with hydrazine base fuels. Both chemically and mechanically, the material is stable and useful as a seal material.

In areas such as long term propellant exposure under compression, both materials are good, with AF-E-124D exhibiting somewhat higher permanent set than AF-E-411 but both being superior to Teflon.

Another elastomer, AF-E-411A was tested during this program. This material is AF-E-411 with an antioxidant added to improve elevated temperature capability. The properties are similar to AF-E-411 but indicate no appreciable improvement and as such is not recommended over the previously mentioned elastomers.

Data are presented for AF-E-124D, AF-E-411 and AF-E-411A related to the following: temperature exposure, air aging, propellant exposure, compression set, stress-strain at various temperatures, compression set (long term and elevated temperature), permeability (gas and propellant) friction and wear.

Comparable data, but in less detail are presented for state-of-the art seal materials; Teflon, Kel-F, Kynar, Vespel, Ethylene Propylene Rubber, Carboxynitroso Rubber and Butyl Rubber and Viton.

A contamination sensitivity investigation plan is presented including a technique for controlled evaluation of relative effects of various configuration contaminant particles.

The seal design procedure outlines a step-by-step procedure which is followed for all seal design applications. It outlines methods to determine whether a particular step should be utilized for the design in question; how to perform that step; and the fail/pass criteria by which the results may be assessed.

A comprehensive bibliography is included of over 200 references related to seal design or seal performance, for information on specific subjects not covered in detail in this report.

3. Program Description

3.0 PROGRAM DESCRIPTION

The program was organized and oriented toward identifying suitable seal materials, developing materials and testing seal materials and designs for extended use with storable propellants. Included in the program were the following activities:

- Survey of sealing technology and assessment of state-of-the-art of sealing
- Determination of critical material and design parameters
- Screening evaluation of candidate materials
- Long-term testing of candidate materials

12.

- Material compounding to improve properties
- Material characterization to determine material properties and characteristics.

The program was organized into five task areas (originally seven tasks before a reduction in scope occurred). These tasks were identified as:

- <u>Task 1 Analysis</u>. This task included a seal technology survey; a determination of critical material and design parameters; and a summary of state-of-the-art in seal technology.
- <u>Task 2 Basic Screening</u>. Designed to perform an initial evaluation of material capability to perform under anticipated Space Shuttle conditions.
- <u>Task 3 Definition and Development</u>. A planning and evaluation phase for material characterization testing.
- <u>lask 4 Material Characterization</u>. Conducting tests to characterize material properties.

Task 5 - Documentation and Reporting Tasks.

3.1 <u>Task 1 - Analysis</u>. As identified above, the basic goals of this task were to survey and assess sealing technology. This was accomplished through a state-of-the-art survey and evaluation of seal design factors. The end

result of this task was an Analysis Report, issued on 6 November 1972; TRW Report Number 72.4781.6-262 entitled, "Analysis Report, Space Shuttle Seal Material and Design Development for Earth Storable Propellant Systems," (Ref. 86). Applicable portions of this report are included as Appendix B.

The sealing technology survey was conducted utilizing resources obtained during a previous program for cryogenic seal development (Contract NAS 9-12500) and augmented with a report on storable propellant applications. The facilities of WESRAC (Western Research Applications Center) were used originally for a comprehensive literature search on seals and seal materials. This was supplemented by a Defense Documentation Center (DDC) literature search, U.S. Department of Commerce (Material Technical Information Service) search, TRW Systems library search; component vendor contacts, and contact with industrial and government sources.

An evaluation of critical seal parameters was conducted under Contract NAS 9-12500 and reported in Reference 88 for cryogenic seals and materials. This evaluation was expanded and revised with respect to storable propellant seal applications and presented in the "Analysis Report" (Ref. 86) for the program.

The "Analysis Report" is a lengthy document since it also includes abstracts of reference documents. As such, excerpts are provided in Appendix B of the major portion.

The following conclusions were reached as a result of the investigations and evaluations during this task (see Appendix B for more detailed information related to these conclusions).

(A) A relatively limited number of polymeric seal materials are available and have been used in the past for storable propellant sealing applications. The most common polymeric seal material by far has been Teflon. Teflon has many advantages and some deficiencies, but in general has been an excellent seal material for most past applications. The use of Teflon has permitted oxidizer applications with N_2O_4 not otherwise possible. The

major deficiencies noted with Teflon seals are the "cold flow" or continuing deformation under load, and a lower tolerance to contaminant particles than elastomers. These and other lesser problem areas may limit cycle life and could compromise a long term, low maintenance concept for Space Shuttle components.

For advanced systems or systems now in fabrication, two new materials have been identified and developed by TRW Systems through contracts with the Air Force Materials Laboratory, Wright-Patterson AFB, and later with NASA Johnson Spacecraft Center, Houston. These materials are:

AF-E-411

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An advanced EPT/HYSTL compound superior in molding and fabrication properties to AF-E-102. It is being used in monopropellant hydrazine spacecraft propulsion systems. Among these are: Atmosphere Explorer (Orbit Adjust Propulsion System) (NASA), Fleet Sat Com (Navy), DSP (Air Force).

AF-E-124D

A cured perfluorinated polymer described in detail in this report for oxidizer service. Being applied to the Delta engine of the Thor-Delta booster.

Other sea! materials commonly in use, both current and past, are:

Carboxynitroso Rubber (CNR)

Oxidizer applications only and relatively limited due to low mechanical properties.

Butyl Rubber

Generally used with fuels only (some limited oxidizer use in past). Has proven satisfactory in fuels but not in common use row because of decomposition of propellant and degradation of elastomer over long term.

Ethylene-Propylene Rubber (EPR)

Superior to butyl rubber for fuel applications.

Kel-F

Used with N_2O_4 oxidizer in limited applications.

Kynar

A thermoplastic used for short term or vapor exposure to fuels $N_2 0_4$, limited application due to lack of long term compatibility.

Ethylene-Propylene Terpolymer/HYSTL

A compound designated as AF-E-102 has been successfully used in monopropellant hydrazine spacecraft propulsion.

(B) Determination of critical seal design parameters tends to be a complex problem as is the design of a specific seal. A general list of critical parameters, rated by degree of criticality was determined to be impractical if not impossible since in the majority of cases it is configuration or application dependent. A summation of the most critical parameters was established as a function of type of seal or function. These are presented in Tables 4-6 through 4-9 of Appendix B.

Critical parameters included:

Inertness
Contamination Sensitivity
Permanent Set
Temperature Effects
Permeation

In specialized cases, surface finishes, valve stroke, sealing load and pressure differential sensitivity were considered critical. Hardness was considered important in each case but not a critical parameter.

3.2 Task 2 - Basic Screening

The purpose of this task was to permit an initial evaluation of material capability and the potential of each candidate material to perform as a seal material. Based on the results of previous programs. (Refs. 89 and 90) the materials selected as candidates were AF-E-124D (for fuel and oxidizer service) and AF-E-411 for fuel service. Modifications of these materials were investigated both during this program and previous programs. Pertinent results are reported and discussed in Section 4 of this report.

The materials investigated during this phase of the program were:

AF-E-411

- AF-E-4îl with 10 parts of Kaydol (a mineral oil to improve low temperature properties)
- AF-E-411 with 20 parts of Kaydol (a mineral oil to improve low temperature properties and reduce compression set)
- AF-E-411 with 1 part of Age-Rite Resin D (an antioxidant to improve elevated temperature)
- AF-E-411 with 3 parts Age-Rite Resin D (added to improve elevated temperature later designated AF-E-411A)

AF-E-124D

Compounding studies of AF-E-124D were conducted previously under Contract NAS 9-12500. The following additives were investigated:

Krytox (a perfluoroether)

Krytox and Teflon Powder

Teflon Powder

DC-440 (a Dow Corning Silicone rubber)

LS-422 (phenylvinyl silicone rubber)

Silene (hydrated silica)

HAF-HS (small particle size carbon black)

SAF (larger structure carbon black)

As a result of this investigation it was concluded that compounding does not appear advantageous in improving any properties of AF-E-124D. The base polymer exhibited the optimum properties.

As part of this task, materials in the form of tensile specimens and compressed 0-rings were placed in long-term storage to obtain as long a period of exposure as possible. The majority of the screening tests were completed early in the program while the long-term testing continued throughout the program.

As a result of this task, three materials were selected for characterization during Task 4. These materials were:

AF-E-411

AF-E-411A (AF-E-411 with 3 parts of Age-Rite Resim D antioxidant resin)

AF-E-124D

3.3 Task 3 - Definition and Development

This task evaluated the results of the screening task and determined the approach to be taken during the characterization task. A matrix for the materials to be tested was developed based on the c. tical parameters and material properties not presently available. This plan was described in the "Material Evaluation and Compounding Plan," Report Number 72.4781.6-284, 20 November 1972.

The tests planned are summarized in Figure 3.1 and include mechanical properties and functional properties.

						Charact	Characterization Data	on Data					
Test		•	,	Afr			N 204			90/20		Other Lest	
Mumber	Title	Material	-100°F	+70°F	+300°F	-100°F	+70°F	+300°F	-100°F	+70°F +300°F	u 5	Conditions	
~	Mechanical Properties	AF-E-411	××	××	××							Load rate 1.0"/min	/min
(e)	Compression Deflection	AF-E-411A	××;	××	××;							Load rate 0.1"/min	/min
	Standard Specimen	AF-E-1240	<××××	××××	****							Load rate 0.31"/min	"/min
(9)	Compression Set	AF-E-411		××						×××		Time 7 days Time 30 days Time 3 months Time 6 months Time 9 months	25-30% Initial Compression
		AF-E-411A		××						***		Time 7 days Time 30 days Time 3 months Time 6 months Time 9 months	25-30% Initial Compression
		AF-E-124D		× ~			** *			× ××		Time 24 hours Time 100 hours Time 2 months Time 3 months Time 5 months	
<u>9</u>	Compression Hysteresis	AF-E-411 AF-E-411A AF-E-124D		 ×××	<u> </u>						1		
<u>[8</u>	Tear Strength	AF-E-411 AF-E-411A AF-E-1240		***									
1(e)	Stress Strain	AF-E-411 AF-E-411A AF-E-1240		×××		<u> </u> 							
. 1(£)	Hardness Swe ¹ ling Wt. Chg.	AF-E-411 AF-E-411A AF-E-124D					×			×××			
1(9)	Propellant Effects	AF-E-411 AF-E-411A AF-E-124D					×			***	×××	Pressure Rise Rate at 200°F	

Figure 3.1 Characterization Task Matrix

		Uther Test Conditions		Static Dynamic Abrasion Static Dynamic	Abrasion Static Dynamic Abrasion	After elevated Lemperature aging			Helium	: Helium Helium	16 RMS Finish -40, +70, +150°F 63 RMS Finish Vary number of cycles Vary exposure time	16 PMS Finish -40, *70, +150°F Vary number of cycles Vary exposure time
		+300°F						_	- *	- ****		
	50/50	+70°F		** **	**				×	× ×	**	×
		-100°F										
n Data		+300°F										
rizatio	N ₂ U ₄	+70°F	<u> </u>		**		_			×		××
Characterization Data		-100°F										
		+300°F	×××	×× ××	××	×××					×	×
	Air	+70°F	×××	****	××××		×<×	×××	×	× ×	××	×
		-100°F	×××	×× ××	××						×	*
		Material	AF-E-411A AF-E-411A AF-E-124D	AF-E-411 AF-E-411A	AF-E-1240	AF-E-411 AF-E-411A AF-E-124D	AF-E-411 AF-E-411A AF-E-124D	AF-E-411 AF-E-411A AF-E-124D	AF-E-411	AF-E-411A AF-E-1240	AF-E-411	AF-E-1240
		Title	Coeff. of Thermal Expansion	Coeff. of Friction 2. Abrasica Tests		Stress- Strain	TGA	Vacuum Effects	Permeability		Seal Load	
		Number	2(4)	2(p)		2(c)	2(4)	2(e)	2(1)		2(9)	

Figure 3.1 Characterization Test Matrix (Continued)

3.4 Task 4 - Material Characterization

The tests planned during the previous task and summarized in Figure 3.1 were conducted during this task. Characterization of the selected materials was performed over a wide temperature range and with the propellants of interest. The details of the tests to be conducted were outlined in TRW Report Number 72.4781.6-284 and are described here rather than include that report, and also to describe tests and peripheral information not provided in that report.

Two basic types of tests were conducted: Mechanical and Physical Properties of Materials; and Parametric Tests. These tests were conducted at ambient temperature in air or propellant as applicable, and at low or elevated temperatures. The propellants used were nitrogen tetroxide as the oxidizer and 50/50 mixture of UDMH and hydrazine usually as the fuel, this being considered generally a worst case condition. The only exceptions to the use of 50/50 is where a larger prior data base has been obtained with N_2H_4 through previous tests and a better evaluation of relative properties would be obtained.

As shown in Figure 3.1, the test parameters were:

Mechanical Properties

- Compression Deflection as a function of load rate and temperature
- Compression set as a function of load time, temperature and propellant exposure
- Compression hysteresis as a function of cycling
- Tear strength as a function of temperature
- Stress strain as a function of temperature and propellant exposure
- Hardness, swelling and weight change due to fluid exposure
- Material effects on propellant

Physical Properties

- Coefficient of thermal expansion
- Coefficient of friction (static & dynamic) against stainless steel in air and propellant

- Stress strain after elevated temperature exposure
- Thermogravimetric analysis in air
- Effect of vacuum exposure
- Permeability to helium and propellant
- Sealing load as a function of surface finish and propellant exposure

In addition, investigation relative to controlled methods of determining contamination sensitivity, and basic effects of contaminants were conducted. These resulted in a possible test technique for determining contamination sensitivity of a material or design. This test plan is described in Appendix C and the work conducted during this program is discussed in Section 5 in conjunction with the Seal Design and Material Guide discussion.

3.4.1 Tests and Test Techniques

Mechanical Properties

Compression - Deflection - ASTM D395 buttons were used for test. In some cases, plied-up slab stock was prepared to conform to the same specimen dimensions. Using an Instron testing machine, the specimens were compressed to about 50% deflection at three decades of loading rate. The load was recorded to allow construction of load vs percent compression plots. These tests were conducted at three temperatures ranging from -100° F to $+300^{\circ}$ F.

Compression Set AMS 3304-214 O-rings were prepared from seal materials and compressed 25% and held for at least three time periods at three temperatures. Measurements were taken before and after test and compression set calculated.

Compression Hysteresis - ASTM D395 buttons or slabs plied-up to the same dimension were compressed using the Instron testing machine to approximately 40% deflection, plotting load/deflection. Rate of loading was the intermediate rate used in the previous compression/deflection tests described above. The sample was repeatedly loaded and unloaded up to 100 times. A plot was made to determine hysteresis and change in load characteristics.

Tear Strength - ASTM D624 Die B specimens were cut from molded sheets of the seal material. These specimens were mounted in an Instron test machine fitted with a constant temperature chamber surrounding the test specimen. Each specimen was conditioned at temperature for at least 20 minutes. At 20-inch per minute crosshead speed, the specimens were tested and the tearing force calculated. The test was performed on triplicate specimens at ambient and +300°F. Low temperature tests proved impractical since specimens would crack rather than tear.

Stress/Strain - ASTM D412 Die D specimens were cut from molded sheets of each seal material. Triplicate specimens were tested at 20-inch per minute crosshead speed. Modulus at 100% elongation, tensile strength at break and elongation at break was recorded for each specimen. Tests were performed at three temperatures ranging from -100° to +300°F, and with specimens after exposure to propellants and to air aging.

<u>Hardness</u>, <u>Swelling and Weight Change</u> - Specimens were exposed to propellants for varying periods of time to determine changes.

Effect of Seal Material on Propellant - Triplicate ASTM D1708 microtensile die specimens were used for this test. These specimens were cleaned with isopropyl alcohol, dried and placed in containers with propellant. Containers were sealed and temperature increased to the maximum feasible for the propellant. Pressures were monitored during the storage period. The propellant was analyzed for non-volatile residue (NVR) and assayed. All data - pressure, NVR and assay was compared with control propellant from the same source of supply.

Physical Properties

Coefficient of Thermal Expansion - The coefficient of thermal expansion of each seal material was measured over the temperature range of -40° to $+300^{0}$ F using a quartz rod dilatometer.

Coefficients of Friction and Wear - The static and dynamic coefficient of friction in air, at low and high temperatures and in propellants were determined with a Dow Corning wear tester. A circular ring which has the seal material moided on it was placed in contact with a stainless steel rider, and using a rotary motion, the coefficients are determined. For

wear determination an oscillatory motion of the ring was used to simulate the reversing action experienced by a ball valve or shaft seal. Wear rate was determined by weight loss as a function of number of cycles.

Stress/Strain After Elevated Temperature Air Aging. ASTM D412 Die D specimens of the mate: ial were stored in a circulating air oven at 300° \pm 10° F for 100 hours. Stress/strain properties were measured after this exposure and compared with data from unaged control specimens.

<u>TGA in Air</u> - Thermogravimetric <u>Analysis</u> (TGA) was measured under adiabatic cenditions with a 3° 5 per minute programmed heating rate. The weight loss (or gain) of the material in air was plotted continuously between ambient room temperature and the temperature at which a severe weight loss occurs.

Effect of Vacuum - Pre-weighed specimens of seal materials were exposed to a vacuum (1.5 x 10^{-5} torr) at ambient room temperature. The samples were weighed after an exposure period of 168 hours.

Permeability - Uncompressed specimens of cured seal material were subjected to helium and propellant permeability testing at ambient room temperature. For hydrazine base fuels, the propellant was placed in the upstream side of the test membrane and a nitrogen sweep conducted the permeated propellant into a glacial acetic acid scrubber. At approxpriate times the acetic acid was titrated with 0.1M perchloric acid to a quinal-dine red end point (pH 3 to 4). Total bases were calculated and a plot of permeability vs. time constructed. For nitrogen tetroxide, a closed system was used with the propellant upstream of the test membrane and a transducer moinitoring pressure in the downstream volume. Using the Gas laws and knowing the dissociation equilibrium of nitrogen tetroxide at the test temperature, the concentration of oxidizer permeating per unit time was determined.

<u>Seal Load Requirements</u> - A commercial valve was modified for use as a seal tester to provide a demonstration of sealing capability and generally verify the characteristics of the selected materials. The seal configuration was used as a simple flat poppet type seal. Seals of both materials (AF-E-124D and AF-E-411) in various test conditions were used. The seal material was used as the seat, with a flat metal poppet as the

Tating element as shown in Figure 3.2. The seal diameter was nominally I inch diameter with an 0.050 inch width. One variable was the poppet surface finish, while others were temperature, propellant exposure and number of cycles.

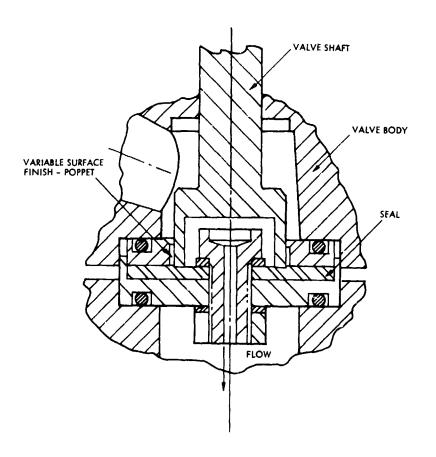


Figure 3.2 Test Valve Seal Poppet Configuration

The test setup is shown schematically in Figure 3.3. Pneumatipressure was used to load the seal, and to determine the seat loading. Gaseous nitrogen was also applied to the seal area with flow being measured with a rotometer type flow meter. The valve was slowly closed until no flow was seen with the flow meter. Leakage flow was then measured with the liquid filled pipette and seat stress increased until no liquid movement in the pipette was seen. This point was defined as "zero leakage" for this test series.

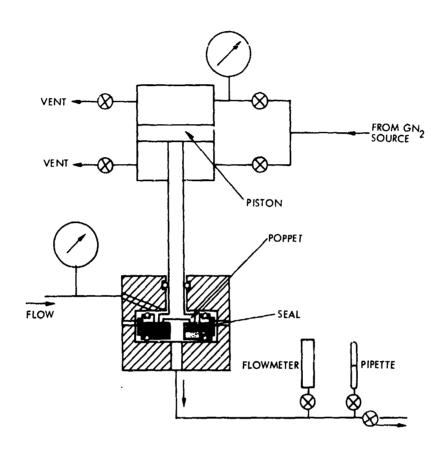


Figure 3.3 Seal Verification Test Setup

4.0 SEAL MATERIAL PROPERTIES

This section presents information on the materials tested during this program, including where applicable, information from other sources. Section 4.1 describes the characteristics of the materials tested (AF-E-124D, AF-E-411 and AF-E-411A). In Section 4.2 the test data are presented by material, and include both the results of the screening tests and the characterization tests. As the test program was structured, many tests were intended to determine the limits of the material as a basis for comparison, and not necessarily reflect intended service conditions. As such, tests for 20 to 100 hours under severe test conditions are meaningful in determining material limitations and modes of degradation, but probably exceed anticipated service conditions.

A compilation of data on other currently used polymeric snals is provided in Section 4.3 and compared to the materials tested during this program in Section 4.4.

Since abbreviations are used throughout the report, a glossary of terms and abbreviations is provided in Table 4-1.

Table 4-1. Glossary of Terms and Abbreviations

Abbreviation	Meaning
T_b	Tensile strength at break in pounds per square inch
E _b	Elongation at break in percent
M ₁₀₀	Stress at 100 percent elongation in pounds per square inch
pli	Pounds per linear inch, (used in tear strength)
Shore	Hardness scale (used for soft materials)
NVR	Nonvolatile residue
TGA	Thermal gravimetric analysis
T_{g}	Glass transition temperature
TŘ-10	Temperature at which a material recovers 10 percent after being stretched and frozen. (ASTM Method D-1329-60)
50/50	A 50 percent mixture of hydrazine and unsymmetrical dimethylhydrazine (50/50 N ₂ H ₄ /UDMH)
g or gr	Grams
CC	Cubic centimeters
Phr	Parts per hundred of Rubber

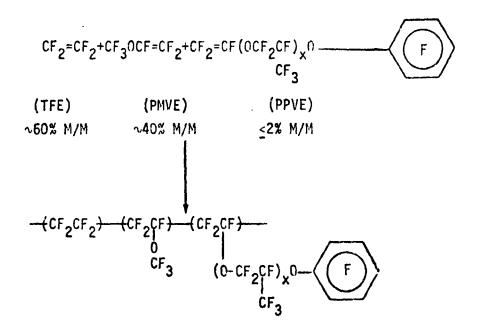
4.1 Descriptions of Materials Tested

Three materials were identified during this program as being improved materials for oxidizer or hydrazine base fuel service. These materials are: AF-E-124D, AF-E-411 and AF-E-411A.

4.1.1 AF-E-124D

AF-E-124D is a material initially supplied by the Air Force Materials Laboratory, Elastomers and Coatings Branch, for use on this program. Subsequently the gumstock was supplied directly to TRW from the manufacturer, the E. I. du Pont Co. Inc. This gumstock is then cured and molded to the final form by TRW.

AF-E-124D is a terpolymer of Tetrafluorethylene, Perfluoromethyl vinyl ether, and a fluorinated monomer used as a crosslinking site. The resulting polymer is fully fluorinated (or perfluorinated). The composition of AF-E-124D is: approximately 60% Tetrafluoroethylene (TFE), approximately 40% Perfluoromethyl vinyl ether (PMVE), and up to 2% Perfluorophenyl vinyl ether (PPVE). Schematically this is shown below:



The gumstock is then cured by a bimolecular condensation of the pendant perfluorophenyl with a potassium alkoxide of polyethylene oxide (PEO):

$$KO(CH_2CH_2O)_nK$$

according to the following cure and post-cure schedule:

Press:

30 minutes/177°C (350°F)

Air Oven:

Room Temperature to 150°C (300°F)

over 4 hours

24 hours/150°C (300°F) 24 hours/177°C (350°F)

24 hours/204°C (400°F)

Raised from 204°C (400°F) to 285°C (550°F) over 6 hours

24 hours/285°C (550°F)

The reaction occurs at the para position of the perfluorinated phenyl ring as shown below:

This results in the cured perfluorinated elastomer designated as AF-E-124D. Each step in the curing process (including molding) is highly critical, with any deviations resulting in a finished product with varying characteristics. To date, the only material designated as AF-E-124D has been made by TRW because of this criticality. The base gumstock may be obtained from E. I. du Pont de Nemours and Co. (du Pont) but should not be designated AF-E-124D until the finished product is characterized and determined to have similar properties to those in this report. A finished product is also provided by E. I. du Pont using the same base gumstock. This product is designated by du Pont as ECD 006. No indication has been made of this material's propellant compatibility, nor is there any intent to do this by du Pont. The purposes for which this commercial grade material is used are other than storable propellant seal applications. The methods and controls used for curing ECD 006 are not available for this report and may be proprietary to du Pont.

In contrast to other fluorinated elastomers which are not fully fluorinated, the perfluorinated elastomers are closely related to the Teflon polytetrafluorethylene (TFE) family of plastics. Chemical inertness and thermal stability of Teflon, combined with the resilience and compliance of an elastomer make the perfluorinated elastomers attractive as seal materials. Flexibility is built into the polymer by the pendant methyl ether groups, without significantly decreasing the chemical resistance of the molecule. The creep or "cold flow" of TFE relative to thermosetting elastomers apparently has been decreased in AF-E-124D by introduction of a relatively reactive monomer into the polymer to provide a site for curing. Linear polymers such as TFE, other thermoplastics and uncured elastomers, are susceptible to stress induced creep as the polymer molecules can slip over one another. Crosslinking reactions develop a three-dimensional matrix of chemically bonded molecules which reduces slippage considerably. This material is also present'y under study at TRW Systems as an elastomer compatible with chlorine trifluoride (u.der Air Force Contract No. F33615-70-C-1514). While the elastomeric properties of this material are not as evident as those of other elastomers such as AF-E-411, the material is

classified as an elastomer and is chemically resistant because of its perfluorinated backbone.

The cure reaction used for AF-E-124D, on the other hand results in covalent linkages which are resistant to creep. While creep can be accommodated to some degree by design, a highly thermoplastic crosslink network is regarded as a serious deficiency for a seal material.

AF-E-124D is a relatively expensive material, but is approximately one-tenth the cost of the only other elastomer potentially useful for oxidizer service, carboxynitroso rubber (described in Section 4.3).

Investigations were conducted with various types of plasticizers and reinforcing agents to improve AF-E-124D mold flow prior to cure, and resistance to creep after cure but the additives were not found to be advantageous. (Much of this work was conducted under a prior contract, NAS 9-12500 "Seal Material and Design Development Program")

Generally, the plasticizers degraded mechanical and chemical properties while reinforcing fillers stiffened the material excessively. Table 4.1-1 summarizes the investigation results. Following this activity, it was determined that AF-E-124D without additives was the optimum material for further characterization tests.

Table 4.1-1. Compounding Studies of AF-E-124D

03-74	436-1	436-2	436-3	4 < -4	436-5	436-6	436-7	435-8
nc ontrol)	Krytox,10	Krytox,10 1-8A,10	T-8A,10	DC-440,10	1,5422,10	Silene D,10	HAF-H5.10	SAF,10
ndard of parison	Best	Poor	Poor	Slight Improve- ment	Very Poor	hery Paar	Worst	Worst
nslu- ent 3%	a) Translu- cont 10%	Translu- cent/ Fibers 9%	Translu- cent 8%	Opaque, Poor Dis- persion 12%	Poor Dis- persion 12%	Poor Dis- persion 8%	Very Rough 81	Very Rough 8≠
		·		J.]			
75	500	2600	-	500	1000	-	-	-
00	3000	2600	2500	800	1100	1506	2800	2000
80	210	100	75	140	110	90	90	90
6	12	8	7	2	3	0	5	5
80	80	90	90	70	€5	90	90	90
	ontrol) indard of parison inslu- ent 3% 75	nontrol) Indiand Best of parison a) Inslucent cont 10% Inslucent 10% Ins	T-8A,10 Poor Poor	T-8A,10 Poor Poor	Translu-cent Cent Poor Poor Slight Improvement	T-8A,10 Poor Poor Slight Wery Poor Improvement Poor Poor Slight Improvement Poor Poor	T-8A,10 Poor Poor Slight Very Poor Poor Improve Poor Poor	T-8A,10

⁽a) Krytox bloc... from the rubber (low stability) upon standing.

4.1.2 AF-E-411

AF-E-411, a peroxide-cured EPT (ethylene-propylene diene terrolymer) compound, was developed under Air Force Contract F33615-71-C-1233 specifically for hydrazine seal service. This material is generally identified as an EPT (ethylene propylene terpolymer)-Hystl (1, 2-2 polybutadiene resin) material from which a number of compounds were previously evaluated for hydrazine service and improved to provide optimum properties, both for service and fabrication. These previous materials were designated as AF-E-71-2 and AF-E-102. AF-E-102 is presently being used in flight applications by the Naval Research Laboratory in their Sol-Rad Satellites.

The composition and cure schedule of AF-E-411 is shown in Table 4.1-2.

Table 4.1-2. Composition & Cure Schedule of AF-E-411

Component	Parts by Weight
Nordel 1635, duPont	100.0
Cah-O-Sil M-5, Cabot Corp.	25.0
B-3000 Resin, Dynachem Corp.;	25.0
Teflon Powder T-8A, duPont	5.0
Zinc Oxide, Baker Reagent	5.0
Calcium Oxide, Baker Reagent	5.0
Vinyl Silane A-172, Union Carbide	1.0
Di-Cup R, Hercules	2.0
Total Parts -	168.0
Cure - 30 Min/350°F	
Postcure - 2 hours/225°F	

Variations of this compound were tested to determine possible improvements in low or high temperature properties. These variations are the results as shown in Table 4.1-3. This table shows the base properties of each material; the effect of exposure to 300°F for 100 hours; and the effect of exposure to hydrazine for 24 hours at 120°F and 72 hours at 160°F, including pressure rise (AP) in a closed container (indicating tendency to decompose hydrazine).

To improve properties at the design goal temperature of +300°F an antioxidant and added. Age Rite Resin D is widely used for peroxide-cured elastomer stabilization and was considered to be non-reactive toward hydrazine. Using mechanical property retention after 300°F air aging as the initial criteria, it was found that this particular antioxidant provided an improvement in AF-E-411's thermal stability.

It was concluded from these tests that the addition of mineral oil (Kaydul) did not improve the low temperature properties through plasticization. Addition of antioxidant was considered beneficial as noted above and retained in the program as a compound designated AF-E-411A.

AF-E-411 has been provided as seal materials for various flight programs including NASA/GSFC Atmosphere Explorer, and Synchronous Meterological Satellite programs.

Table 4.1-3. Variations and Properties of AF-E-411

(Composition)	.ar-E-411)	(+10 phr kaydol 0:1)	(+20 phr Kaydol 0:1)	(+1 0 phr Antioxident)	(+3.0 phr Artio+1, /1)
M ₁₀₀	1650	1650	940	เลนปี	1325
1 _B	2200	2600	2100	2300	2500
£ E	150	170	190	130	220
Set	3	3	4	3	6
Shore A	89	89	86	90	89
Tear	170	280	205	320	280
Air Aged 100h/300°F	T				
				1850	1425
M100	800	1200	725	2000	2300
T _B	50	50	50	110	180
E _B Set	0	0	0	3	6
Shore A	93	93	93	91	90
Tear	130	115	105	230	285
24n/120°F + 72h/160°F					
	2100	1250	1250	2100	1600
^M 100	2400	2250	1950	2600	2100
, R	140	190	190	140	160
f _B Set	3	6	6	5	5
Shore A	90	89	88	91	90
/P, psi	21.4	20.0	20.0	20.5	20.5
(.ontro) = 19 4)		1			j

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4.1.3 AF-E-411A

This compound is one variation of AF-E-411 which indicated potential improvement through the use of additives. Using 3 parts of Age-Rite Resin D, it was determined that after 300°F exposure, improved properties (as compared to AF-E-411) resulted. In all other respects the formulation of AF-E-411A is identical to AF-E-411.

The composition of AF-E-411A is shown in Table 4.1-4.

Table 4.1-4. Formulation of AF-E-411A

Component	Par	ts by Weight	
Extracted Norde! 1040 EPT		100.0	
Cab-O-Sil M-5		25.0	
HYSTL B-3000 Resin		25.0	
T-9A TFE Powder		5.0	
Zinc Oxide		5.0	
Calcium Oxide		5.0	
Vinyl Silane A-172		1.0	
Di-Cup R		2.0	
Age-Rite Resin D		3.0	
	Total Parts	171.0	

4.2 Properties and Characteristics of Tested Materials

The test data presented here are only for those seal materials tested during this program, namely AF-E-124D, AF-E-411 and AF-E-411A. These data include screening and characterization test information. Some information is provided, for completeness, from other programs or sources and is identified as such.

4.2.1 AF-E-124D

It is desirable for many reasons to use the same seal material for both fuel and oxidizer. The only material identified through the earlier phases of this program with this capability besides Teflon is AF-E-124D.

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Mechanical properties of AF-E-124D in air are shown in Table 4.2-1 and cover the anticipated Space Shuttle temperature requirements. The mechanical properties are good for ambient and low temperatures, with the expected increase in tensile strength and reduction in elongation at -100°F. At 300°F the material properties are appreciably reduced indicating limited application at this temperature, for any period of time.

Air aging at $+300^{\circ}\text{F}$ and returning to room temperature produces no effect upon AF-E-124D because it is post-cured in an air oven for 24 hours at 550°F. The data in Table 4.2-3 indicate that even after $+400^{\circ}\text{F}$ exposure the material is not degraded (within experimental error). Immersion of AF-E-124D in $50/50~\text{N}_2\text{H}_4$ -UDMH for three months at room temperature also had no effect upon mechanical properties (Table 4.2-4).

Table 4.2-3 reports the effect of nitrogen tetroxide on AF-E-124D after 7 days, three months, 6 months and 9 months at room temperature; and after 100 hours at +160°F, and 7 days at 200°F. The property change due to ambient room temperature exposure (+65°F to +80°F) indicates relatively little property change, the 6 month change in tensile strength being a reduction of 18%, and at 9 months an apparent reduction of only 10%. Since there is no mechanism whereby an increase in strength should occur with propellant exposure, this must be attributed to test variability. It can be concluded however that the tensile strength must be approximately equal to the 6 month value. As the temperature is increased, a more pronounced change is seen with 100 hours at 160°F causing a tensile strength reduction of 24% and 7 days at 200°F a reduction of 49%. This table also indicates the effect of 50/50 on AF-E-124D properties at room temperature. A larger change at room temperature is seen, a maximum of 21% reduction in tensile strength at 6 months. At elevated temperature a reduction of 30% in tensile strength occurs after 7 days at 200°F.

In Table 4.2-4 the dimensional and weight change in AF-E-124D due to exposure to N_2O_4 or 50/50 are shown. Considerable swell is experienced in N_2O_4 while very little increase is noted in 50/50. After a 7 day outgassing period, the AF-E-124D returns to essentially its initial condition.

In Table 4.2-5 the tendency for AF-E-124D to decompose hydrazine is indicated. No significant decomposition occurs at room temperature. At +200°F a pressure rise of 11.2 psi above the 50/50 control was noted, which is significant. At 200°F the pressure in the control propellant (50/50) reached 28.5 psi. The 50/50 pressure is approximately the vapor pressure of 50/50 at 200°F, and the resulting test pressures were considered the safe limit of the glass test apparatus used. Although similar tests were run with N_2O_4 at 200°F, no useful information could be obtained since the pressure rise is strongly a function of NO_2 concentration and many conditions can affect the N_2O_4 — NO_2 equilibrium. The mechanical properties data from these specimens are shown in Table 4.2-3.

This would indicate the prolonged exposure at +200°F to hydrazine base propellant will result in some propellant decomposition when in contact with the seal material. If the exposed area is small, as in a valve seat or static seal, this should have essentially no system effect.

The effects of various common cleaning solvents are summarized in Table 4.2-6. As indicated in this table little permanent effect is exhibited as the result of 14 day exposure to isopropyl alcohol, distilled water, or trichlorethylene. Freon TF results in appreciable mechanical property and dimensional changes. This is a typical effect experienced with many elastomeric materials. It is recommended that Freon TF not be used with AF-E-124D.

Figure 4.2-1 shows the compression vs deflection characteristics of AF-E-124D at +70°F and +300°F and two loading rates. The resulting curves indicate that the parameter is relatively independent of load rate. 55% was selected as an arbitrary maximum value. The specimens did not fail at this compression. A typical seal deflection of 25% would require about 500 psi 1 ·1, while at +300°F the load is only about 200 psi. Figure 4.2-2 shows that at -100°F for the same deflection

approximately 6000 psi is required. Repeated loadings at ambient room temperature to 30% and 50% deflection (Figure 4.2-3) shows that the material is very resistant to hysteresis losses under these conditions. The difference in load at 25% deflection between cycle 1 and cycle 100 is very small.

Compression set characteristics in air of various configurations are shown in Table 4.2-7. This table is a compilation of a number of tests categorized by temperature.

From this table, it is obvious that configuration is very important in the degree of compression set which occurs. In general, a large mass such as a 1" dia x 1/2" high button experiences less set than an 0-ring. The other, and most important factor, is time. In an unconstrained specimen such as these, an increase in set occurs as a function of time. In a constrained specimen such as an 0-ring in a properly designed gland, or a stopped valve, the load would decrease as set occured and the permanent set would level out after a short period of time.

In this report, compression set is reported by two calculation methods. Each method is useful for specific purposes.

Method A is used in determining degree of compression for static seals and is considered to be of most value in establishing sealing capability. Method B is a more standard ASTM method which is usually referenced in the literature, and results in a higher indicated value. The methods of calculation are:

$$\frac{\text{Method A}}{\text{\% Compression Set}} = \frac{t_i - t_f}{t_i} \times 100$$

Method B

% Compression Set =
$$\frac{t_i - t_f}{t_i - t_c} \times 100$$

Where: t_i = initial thickness

t_f = final thickness

t_c = compressed initial thickness

Compression set of AF-E-124D after various exposures to hydrazine base fuels and N_2O_4 is shown in Table 4.2-8. At moderate temperatures the compression set is low (approximately 15%) even for long term as identified by the 9 month data. As temperature is increased for an extended period the compression set increases, which is related to the reduced tensile properties identified in Table 4.2-1 as temperature increases. Short elevated temperature exposure times would not appear to be detrimental to the sealing capability. Figure 4.2-4 shows an AF-E-124D 0-ring after being exposed to $\mathrm{N}_2\mathrm{O}_4$ at room temperature in a compression test fixture for a two month period. No degradation is apparent an: the original flash on the I.D. remains, indicating no attack even in thin sections. (The O-rings were not deflashed prior to testing.) Also in Figure 4.2-4 are shown two 0-rings which were exposed to N_2O_A at 200°F for 22 hours, while compressed at 30%. Some deformation occurred but no marked physical deterioration was noted. In specimen 2 the O-ring was mechanically separated. The cause of this failure is considered to be the type of test fixture used. The O-ring is compressed without annular support such as would be provided in an O-ring groove. At elevated temperature the tensile and tear strength is reduced (See Table 4.2-1) and the O-ring is sheared more readily. Without groove support, the shearing action continues until physical separation occurs. The 200°F/22 hour point appears to be the maximum time-temperature combination where a slight difference in loading or characteristics are amplified. The two O-rings were tested simultaneously under theoretically identical conditions.

Also in Figure 4.2-4 is shown an AF-E-124D 0-ring after 200°F/22 hour exposure to 50/50 while at 30% compression. As shown here and indicated by the data, property retention was good. Figure 4.2-5 indicates the degree of resilience remaining after compression and the recovery rate after release. As a typical elastomer the maximum recovery occurs rapidly (within one hour). The N_2O_4 curve in Figure 4.2.5 shows an upward trend as a function of time after release. This apparent increase in compression set is due to the loss of volume swell experienced during exposure to N_2O_4 . This results in a lower thickness specimen as the N_2O_4 evaporates. While no data were obtained between 1 hour and 24 hours, the curve is expected to

approach a maximum value rapidly and then remain essentially constant. This swell and contraction characteristic indicates that a desirable condition would be to have AF-E-124D exposed to propellant continuously once initial exposure has occurred.

Table 4.2-9 presents some thermal characteristics of this material. The TR-10 temperature (retraction temperature) is the temperature at which a stretched frozen elastomer specimen regains its "rubbery" character in the absence of imposed work. This test is done by stretching a specimen to a predetermined length, and freezing it at that elongation. The specimen is gradually warmed and the temperature where contraction reaches 10% is defined as the TR-10 temperature (ASTM D1329-60). The significance of the TR-10 temperature, or the glass transition temperature (that temperature where a material undergoes a transition to become hard and rigid), as relates to sealing capability is not established. Theoretically, the material is brittle below this point, but as indicated in the -100°F compression deflection curves, it is resilient and earlier testing (Refs. 89 and 90) have shown effective sealing capability for AF-E-124D in liquid nitrogen (-320°F) and liquid hydrogen (-423°F). By comparison, the TR-10 value for Teflon which is used as an effective low temperature sealant, is approximately +77°F. The liquid oxygen impact resistance is also indicated in this table with no detonations in the standard ABMA test series.

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The Thermal Gravimetric Analysis (TGA) values are significant in that they represent the temperature of extensive material degradation through weight loss. A 1% weight loss of AF-E-124D is experienced at +761°F, and a 50% weight loss occurs at 868°F. This indicates that at any temperature below +760°F, loss of material through thermal decomposition is minimal.

Permeability coefficients for helium, N_20_4 and 50/50 are shown in Table 4.2-10. Rate of permeation from test data is shown in Figure 4.2.6.

The typical permeation curve has an induction period and reaches equilibrium after a given period of time. It is the linear portion that determines the constant, P. As indicated in the table, the units of P are:

$$P = \frac{(Scc) (cm)}{(cm^2 (sec) (cm Hg))} = \frac{(total permeation) (thickness)}{(area) (time) (pressure)}$$

$$= units \times 10^{-10}$$

Coefficient of Friction and Abrasion

Coefficient of friction ($f_{\rm C}$) measurements were made using the Dow Corning ALPHA model LFW-l friction and wear tester shown in Figure 4.2.7. Measurements were made on the three seal materials in air and propellant. The air measurements were made at ambient temperature, +270°F and -110°F using two velocities for dynamic $f_{\rm C}$ data. Measurements in propellant were made at ambient temperature using only a single velocity. Both air and propellant measurements were made five times to minimize data anomolies. The rider blocks were made from 300 series stainless steel as specified by Dow Corning. The test rings and rider blocks are shown in Figure 4.2.8.

To simulate expected seal surface finishes, the elastomer finish was approximately 2 RMS and the rider approximately 8 RMS.

The coefficients of friction for varied temperatures in air, are shown in Figure 4.2.9 as a function of surface speed. Figures 4.2.10 and 4.2.11 provide friction coefficient data in N_2O_4 and 50/50. The coefficient of friction of AF-E-124 D is unexpectly low, both static and dynamic. This material feels somewhat sticky to the touch and tends to stick to itself, but this characteristic does not appear to influence the friction characteristics.

figure 4.2.12 shows wear as a function of number of cycles, compared with Teflon and AF-E-411.

This test was conducted using an oscillatory reversing motion of approximately one inch. In this way, the test simulates the reversing actions of a ball or butterfly valve seal, or the reciprocating action

of a shaft seal. The tendency for the seal surface to be oriented in a preferred direction is eliminated and probably is a worst case condition. As seen from this curve, this material is not a high life material in a direct sliding application. Although the quantitative weight loss is not easily correlated the relative weight loss as compared to Teflon or the other elastomers can be used.

Miscellaneous Properties

Other parameters determined during this program include specific gravity, effect of vacuum exposure and impact strength. These parameters are summarized in Table 4.2.11. Of particular importance is the lack of dimensional or weight change as a result of vacuum exposure. Included in the test samples was a ball valve seal of the configuration used in the TRW LMDE ball valves to determine any vacuum effects on complex shapes. A vacuum of 1.5×10^{-6} torr was maintained for 168 hours. The values reported are the average test values of a number of specimens and although some positive and negative changes were indicated, the values are considered zero, within test accuracy.

Also shown in Table 4.2-11 is the mold shrinkage characteristic of AF-E-124D. This value of 12% is very high and creates problems in molding, particularly where high precision is desired. However, the major problem is in mold design, and generally requires a few iterations to obtain proper mold design. As an example of the complex shapes which can be molded is the ball valve seal used in the vacuum effects test. This AF-E-124D seal is shown in Figure 4.2.13, and shows that excellent results can be obtained, even with a complex part.

Valve sealing verification tests were conducted with a modified commercial valve used as a test fixture, as described in Section 3.4. This fixture used a flat disc type seal as the seat, with a flat mating popper. The sealing diameter was approximately 1 inch diameter with an 0.050" sealing width. This series of tests was conducted to provide verification of sealing capability and for comparison rather than as design data. "Zero leakage" as defined for these tests is no gaseous nitrogen leakage as measured by the water displacement method described in Section 3.4. Figure 4.2.14 indicates AF-E-124D and AF-E-411 seat

stress required to obtain threshold sealing as a function of temperature. As expected, a relatively high stress is required at -50°F but remains relatively constant up to +150°F. In Figure 4.2.15, the effect of cycling on maximum seal stress is shown after exposure to propellant. AF-E-124D indicates little change up to 200 cycles, while AF-E-411 shows a slight decrease. The range of stresses are essentially a constant value at these low (\sim 300 psi) stress levels. Figure 4.2.16 shows the effects of various propellant exposure periods on minimum sealing stress. In 50/50, both AF-E-124D and AF-E-411 are essentially constant. In N₂0₄, AF-E-124D shows a slight reduction, probably caused by swelling which softens the seal somewhat.

Table 4.2-1

Mechanical Properties of AF-E-124D

In Air at Various Temperatures

Parameter	Test Temperature	Value
T _b , psi	-100°F	3100
E _b , %	-100°F	25
Tear, pli	-100°F	70
M ₁₀₀ , psi	+ 75°F	925
T _b , psi	+ 75°F	2150
E _b , %	+ 75°F	205
Tear, pli	+ 75°F	180
Shore A	+ 75°F	86
T _b , psi	+160°F	900
M ₁₀₀	+160°F	275
Ep	+160°F	155
Tear	+160°F	100
M ₁₀₀ ,psi	300°F	200
T _b , %	300°F	250
E _b , %	300°F	110
Tear, pli	300°F	10

Table 4.2-2
Effects of Air Aging on AF-E-124D Mechanical Properties

Hours Aged	Temperature (°F)	Media	Property	Retention of Property
100	+400°	Air	M ₁₀₀ T _b E _b Shore A	100%100%100%+ 2 Change

Table 4.2-3

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AF-E-124D Mechanical Properties After Propellant Exposure

	PARMETER			:		*	f Propert	X of Property Retained				
590 psi 86% 1975 psi 96% 200% 100% s) +0(change)			N ₂ 0 ₄ (1)	50/50 ⁽²⁾	N204 (2)	N204 (3)	N204 (A)	50/50(4)	N ₂ 0 ₄ (5)	50/50(5)	N ₂ 0 ₄ (6)	50/50(6)
1975 psi 96% 88% 89% 76% 82% 79% 51% 70% 90% 200% 100% 95% 100% 140% 97% 97% 180% 196% 100% 7 ±0(change) +2 -5 -2 0 +3 -18 -3 -2	* 100	590 ps i	86%	100%	93%	85%	368	216	36%	808	94%	105%
200% 100% 95% 100% 140% 97% 97% 180% 196% 100% 7 ±0(change) +2 -5 -2 0 +3 -18 -3 -2	- 4	1975 psi		88%	89%	76%	82%	762	51%	70%	% 06	102%
7 ±0(change) +2 -5 -2 0 +3 -18 -3 -2	م س	200%	100T	326	100%	140%	2/6	378	180%	196%	100%	100%
	Shore A (points)	٦.	±0(change)	+5	9		0	 £	-18	en en	-2	+5

(1) 7 Days @ R.T.* and Air Dry 7 Days at R.T.

(2) 3 Months @ R.T. and Air Dry / Days at R.T. (3) 100 Hours @ 160°F and Air Dry at R.T. for 7 Days

(4) 6 Months @ R.T. and Air Dry 7 Days at R.T. 7 Days @ 200°F and Air Dry 7 Days at R.T. (2)

(6) 11 Months @ R.T. and Air Dry 7 Days at R.T.

*R.T. - Room iemperature (70-75°F)

Table 4.2-4

At-E-124D Weight and Dimensional Changes

Due to Propellant Exposure (1)

N.	204	50)/50
3 Month	11 Month	3 Month	11 Month
+10.4%	12.5%	0.6%	1.3%
+ 3.0%	5.0%	~ 0	1.2%
+ 5.7%	4.0%	1.0%	0.6%
+ 2.3%	2.0%	~ 0	3.6%
18.0%	13.0%	3.0%	1.8%
7.0%	6.2%	c	1.8%
	3 Month +10.4% + 3.0% + 5.7% + 2.3%	+10.4% 12.5% + 3.0% 5.0% + 5.7% 4.0% + 2.3% 2.0% 18.0% 13.0%	3 Month 11 Month 3 Month +10.4% 12.5% 0.6% + 3.0% 5.0% 0 + 5.7% 4.0% 1.0% + 2.3% 2.0% 0 18.0% 13.0% 3.0%

- (1) Exposure at ambient room temperature (70^{-75})
- (2) Immediately upon removal from propellant

Table 4.2-5
Effects of AF-E-124D on 50/50 Propellant

Exposure Time (Hours)	Exposure Temperature (°F)	Fropellant	Pressure Rise Abova Control(PS	1)
72	20G°F	50/50	+11.2 ^(a)	a) Surface area of material = 32 Cm ² Volume of propellant = 25 Ml Ullage = 71 Ml Glass container

Table 4.2-6

AF-E-124D Mechanical Properties After Cleaning Fluid Exposure
(14 Pays at Ambient Room Temperature and Air Dry for 7 Days)

		Fluid		
Property	Freon TF	Trichloethylene	Distilled Water	Isopropyl Alcohol
M ₁₀₀ , psi	-56%	- 6%	+12%	+ 5%
T _b , psi	-70%	-17%	<u>+</u> 0	+15%
E _b , %	+12%	+10%	+12%	+13%
Swelling, %	+20%	+10%	<u>+</u> 0	<u>+</u> 0
Weight Change, %	+40%	+ 1%	<u>+</u> 0	<u>+</u> 0
Shore A	- 7	+ 8	+ 6	+ 8

Table 4.2-7

Compression Set in Air - AF-E-124D

				% Compres	sion Set
Temperature °F	Time Hours	Configuration	% Initial Compression	After Release	After 24 Hours
77	24	Button	30	19.2	2.6
77	100	Button	23	36.5	11.8
160	720	0-Ring	25	65	
160	24	Button	23	69	58.5
200	24	Button	27	71	61
400	336	0-Ring	25	30.4	
400	456	0-Ring	25	52	***
400	456	0-Ring	25	36	
400	456	Button	25	12	
400	22	Laminates	25	18.1	
400	22	0-Ring	25	17.9	
400	22	Button	25	14.1	
400	648	0-Ring	25	36	

% Compression Set =
$$\frac{t_i - t_f}{t_i - t_c} \times 100$$

t_i = Initial Thickness

 $t_f = Final Thickness (Immediately on release)$

 t_c = Thickness of Compressed Sample

Configurations

Button 1" dia. x 1/2" high

Laminates - .060 discs l" dia plied to 1/2" height

O-Ring - Standard -214 O-Ring (U.1250 inch nominal cross section)

Table 4.2-8

AF-E-124D Compression Set in Propellants (0-Ring Test Specimens)

Temperature °F	Time Hours	% Initial Compression	Propellant	Compress A	sion Set (1)
75	1440 (2 months)	25	50/50	13.5	54.3
75	1440 (2 months)	25	N ₂ 0 ₄	11.0	38.5
75	2160 (3 months)	30	50/50	11.0	38.5
75	6480 (9 months)	30	50/50	16.9	59.1
75	6480 (9 months)	30	N ₂ 0 ₄	15.3	53.5
160	696	30	N ₂ 0 ₄	23.5	82.3
200	22	25	N ₂ H ₄	22	77
200	22	25	UDMH	24	84
200	22	25	ММН	25	87.5
200	22	30	UDMH	20	70
200	22	30	N ₂ O ₄	14.8	52

(1) Method A - % Compression =
$$\frac{t_i - t_f}{t_i}$$
 X 100
Where t_i = Initial Thickness = .1250 (nominal)
 t_f = Final Thickness - Immediately upon release
Method B - % Compression = $\frac{t_i - t_f}{t_i - t_c}$ X 100

Where t_c = Thickness of Compressed Sample

Table 4.2-9
Thermal Properties of AF-E-124D

PARAMETER	PROPERTY VALUE	
TGA ⁽¹⁾		
1% weight loss	761°F	
50% weight loss	868°F	
TR-10 ⁽²⁾	+ 37°F	
Coefficient of Thermal	458	
Expansion (Linear) 1M/1N/°F x 10 ⁻⁶	$(+60^{\circ}F \text{ to } +342^{\circ}F)$	
Glass Transition Temp. (approx.)	+ 37.5°F	
LO ₂ /Impact Sensitivity ⁽³⁾	0/20	

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- (1) Temperature at which indicated weightless occurs.
- (2) Temperature at which elastomeric properties reappears after being frozen at lower temperatures (ASTM D-1329-60)
- (3) ABMA Test 72 ft-1b impact load number of reactions/total number of tests.

Table 4.2-10
Permeability Coefficients for AF-E-124D

		UDMH, 70°F 1 Atmosphere	N ₂ O ₄ 70°F 1 Atmosphere	N ₂ H ₄ , 70°F 1 Atmosphere
$P = \frac{(scc) (cm)}{(cm^2) (sec) (cmhg)}$	100 x 10 ⁻¹⁰	0 (22 days)	80 x 10 ⁻¹⁰	0 (42 days)

Table 4.2-11
Miscellaneous Properties of AF-E-124D

	Miscerianeous Propercies		
	PARAMETER	VALUE	
,	Specific Gravity Vacuum Effects ⁽¹⁾	2.05g/cc	
	<pre>% Dimension Change % Weight Change</pre>	+0.7 to -0.9* +0.4 max.*	
	Mold Shrinkage	∿ 12%	

(1) After exposure for 168 hours torr

(*Note: Considered to be 0 within test accuracy)

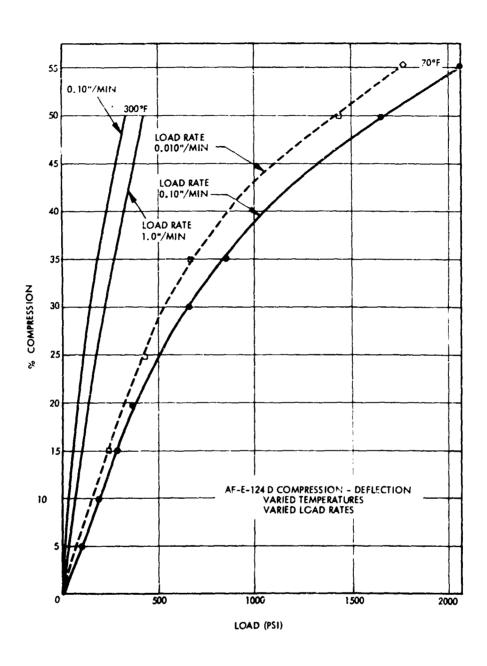
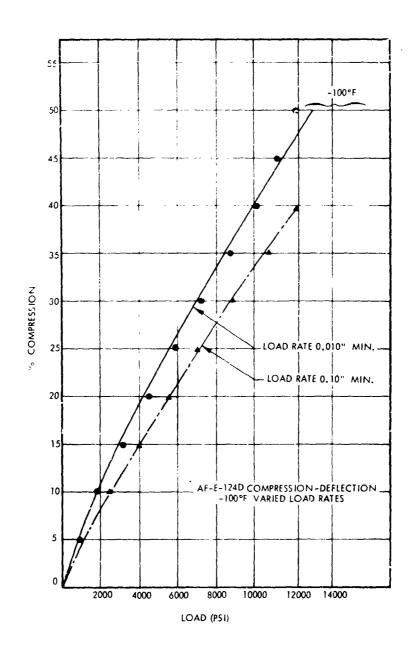


Figure 4.2.1 AF-E-124D Compression Deflection Characteristics at 70°F & +300°F (ASTM D395 Specimens)



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Figure 4.2.2 AF-E-124D Compression Deflection Characteristics at -100°F (ASTM D395 Specimens)

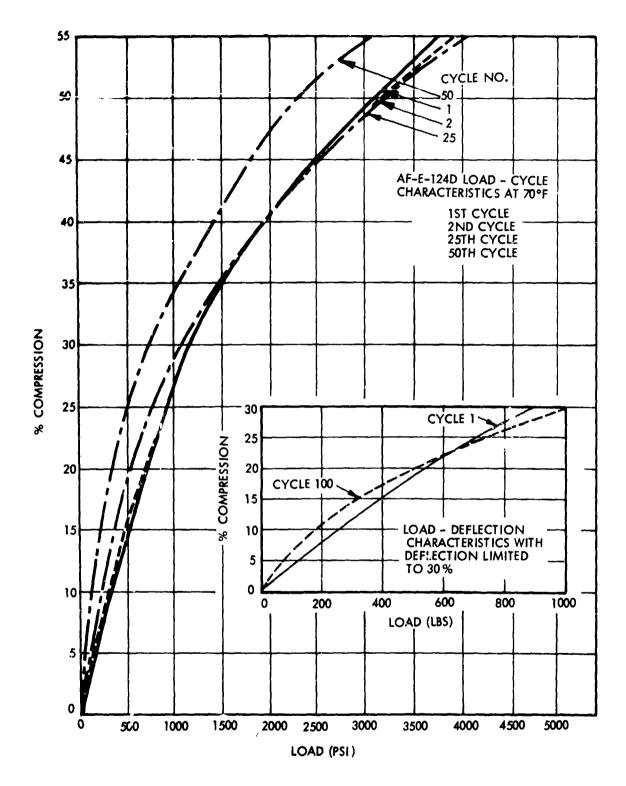
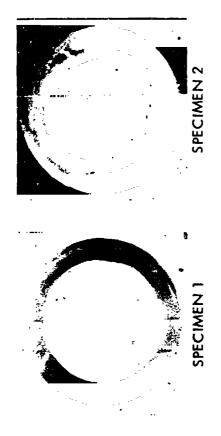


Figure 4.2.3 AF-E-124D Load-Cycle Characteristics at 70°F (ASTM D395 Specimens)



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AF-E-124D O-RINGS AFTER 200°F/22 HOUR N₂O₄ EXPOSURE AT 30% COMPRESSION (1.5X)
(NOTE MECHANICAL PEROPMATION PESHITING EP

2-MONTH EXPOSURE TO N2O4

ROOM TEMPERATURE 30% COMPRESSION

(1.5X)

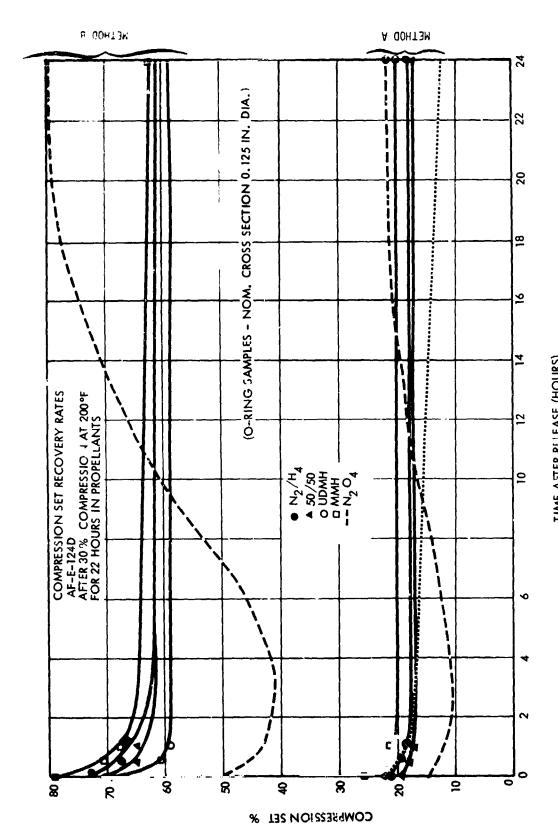
(NOTE MECHANICAL DEFORMATION RESULTING FROM CLAMPING FIXTURE NOT PROPELLANT EXPOSURE)

(S)

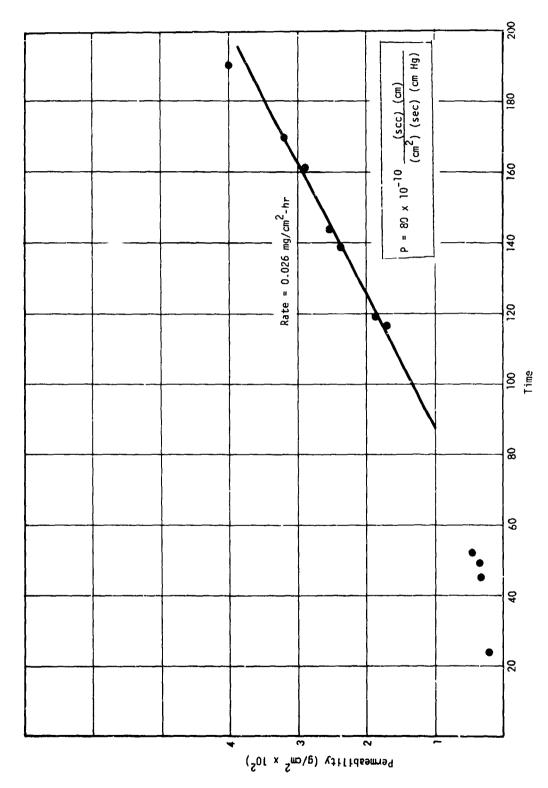
(NOTE ORIGINAL FLASH REMAINS ON I.D.)

AF-E-124D O-RING AFTER 200°F/22 HOUR 50/50 EXPOSURE AT 30% COMPRESSION (1.5X) (MOLD LINE VISIBLE)

Figure 4.2.4 AF-E-124D O-Rings After Exposure to Propellants



TIME ATER RELEASE (HOURS)
Figure 4.2.5 AF-E-124D Comp ession Set Recovery Rates After 30%
Compression at 200°F for 22 Hours in Propellants



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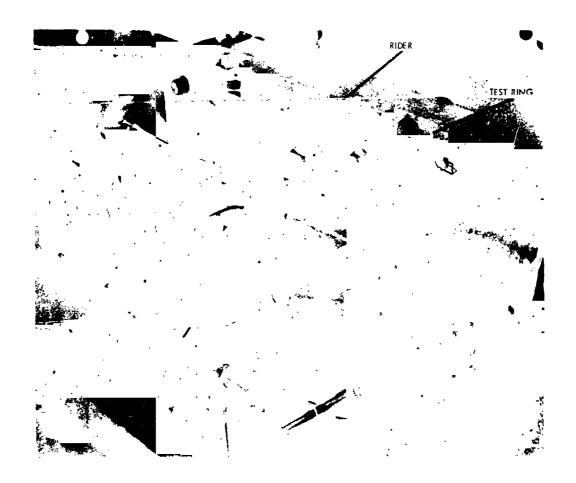


Figure 4.2.7 Dow Corning Wear Tester



Figure 4.2.8 Wear Ring and Rider

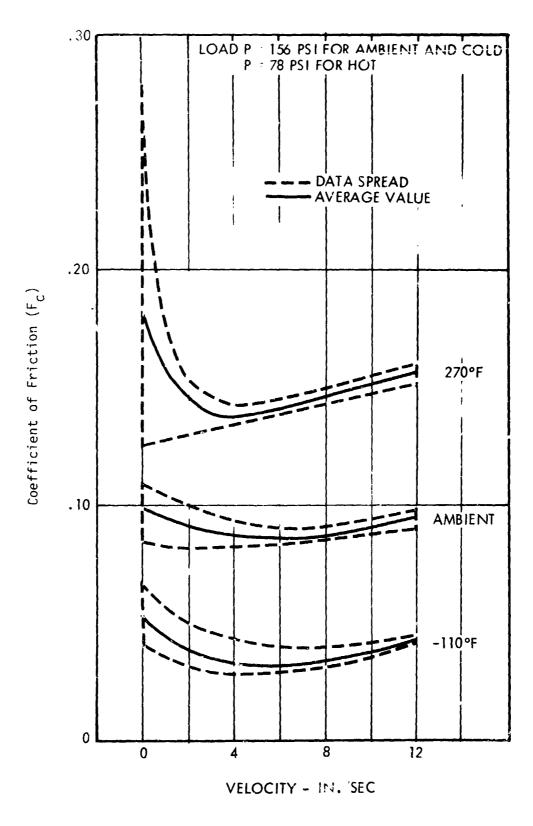


Figure 4.2.9 AF-E-124D Coefficients of Friction in Air

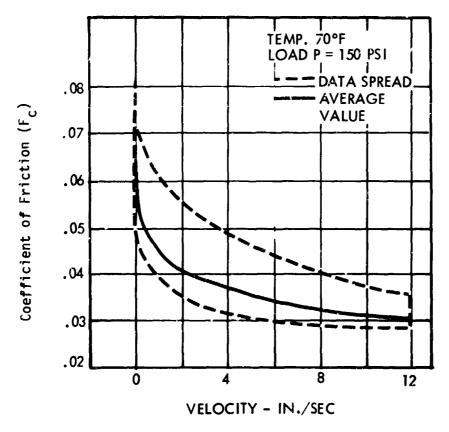


Figure 4.2.10 AF-E-1240 Coefficient of Friction in 50/50

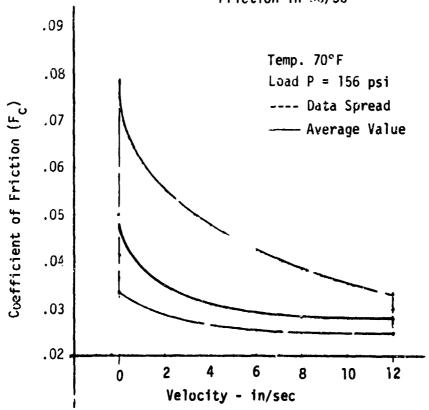


Figure 4.2.11 AF-E-124D Coefficient of Friction in N_2O_4

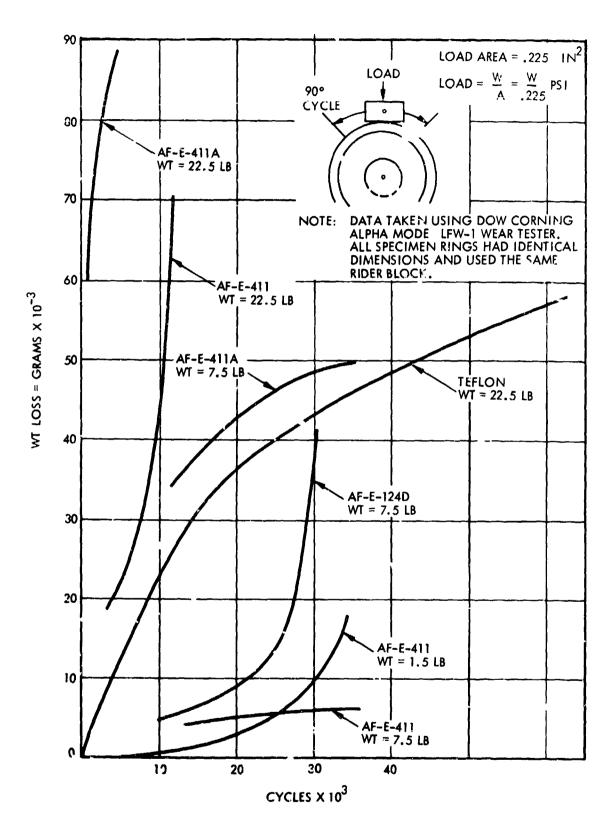
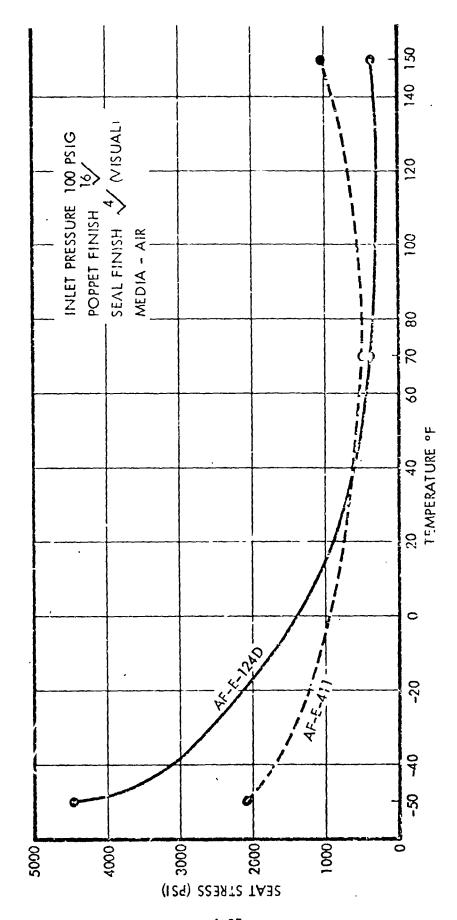


Figure 4.2.12 Wear Comparison of Teflon and AF-E-411, AF-E-411A, and AF-E-124D



Figure 4.2.13 A: 124D Ball Valve Seal (LMDE Shutoff Valve Design)



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Figure 4.2.14 Seat Stress to Obtain Zero Leakage* As a Function of Temperature (*Zero Leakage - Nc GN₂ Leakage Measured by Water Displacement)

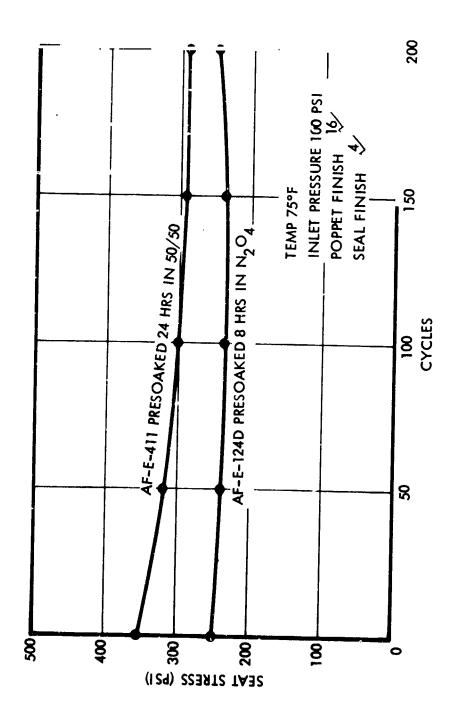
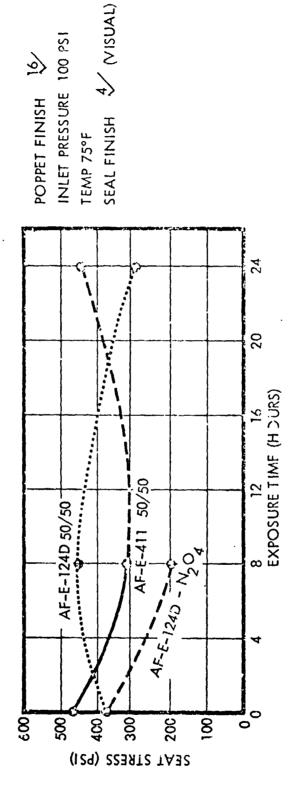


Figure 4.2.15 Seat Stress to Obtain Zero Leakage* As a Function of Number of Cycles (*Zero Leakage - No GN₂ Leakage Measured by Water Displacement Method)



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Figure 4.2.16 Seat Stress to Obtain Zero Leakage* As a Function of Propellant Exposure Time (*Zero Leakage - No GN₂ Leakage Measured By Water Displacement)

4.2.2 AF-E-411

AF-E-411 is intended only for fuel service and as such is characterized for this application. As with AF-E-124D it was selected as the most suitable material from screening tests and previous test history.

Mechanical properties are reported in Table 4.2-12 for AF-E-411 in air at temperatures over the expected range of Space Shuttle requirements. At room temperature, AF-E-411 is characterized as a relatively high modulus materia; because of a high crosslink density. Of the three materials, AF-E-411 best retains high tensile strength over the range of test temperatures.

Table 4.2-13 shows the effect of 300°F air aging on the mechanical properties of AF-E-411. This property is perhaps the weakest of those tested. After 100 hours at 300°F, the room temperature stress-strain properties of AF-E-411 were about one-third the original values. Air, and more correctly the oxygen in the air, attacks EPDM polymer at elevated temperature. Polymer chain scission is the primary degradation mechanism.

After propellant exposure at room temperature and up to 160° F, AF-E-4ll shows little sign of degradation, as shown in Table 4.2-14. Original mechanical properties of the material are shown and in comparison with the initial values the particular batch used for these tests shows slight undercure (the reason for this is unknown). After aging in the propellants, AF-E-4ll provides properties in excellent agreement with the initial values. There is experimental error of approximately $\pm 10\%$ due to mechanical property measurement indicating that the material is not significantly degraded by the propellants at temperatures up to 160° F or as a result of long-term immersion in propellant at room temperature.

In applications, when large seal areas have contact with the propellant during long storage periods, it is important to know if the seal degrades the propellant. In the case of hydrazine base fuels, the two most accessible forms of degradation are pressure rise (caused by hydrazine decomposition products) and an increase in the propellant's

non-volatile residue (NVR). Table 4.2-15 shows that AF-E-411 does not significantly decompose hydrazine. However, the propellant NVR was increased by 100 hour/160°F storage with the rubber. The NVR material had an oily appearance, and was insoluble in dilute hydrochloric acid, suggesting that it was organic. Light oils are present in peroxide-cured EPDM and these possibly were extracted by the propellant. Compatibility data from other research programs and from flight qualification tests have provided NYRs lower than the results reported here, suggesting that the particular lot of MIL-SPEC hydrazine may also influence the NVR value.

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Table 4.2-16 shows the effects of immersing AF-E-411 in representative cleaning fluids. Isopropyl alcohol is the recommended cleaning solvent, while distilled water also has no effect. Freon TF and trichloroethylene are not recommended since high permanent weight and dimensional changes result. This is typical of the effect of these solvents on elastomers.

Compression vs deflection curves for AF-E-411 are shown in Figures 4.2.17 through 4.2.20. Figure 4.2.17 shows that, over two decades of loading rate, at ambient room temperature, the material has reasonably constant loading characteristics. For a 25% compression, the material requires approximately 1400 psi. At +300°F (Figure 4.2.18) the material is also insensitive to the loading rate over two decades. The material can also be compressed to 55% without failing. At this temperature, a 25% compression requires approximately 1000 psi. Figure 4.2.19 shows the load characteristics at -100°F. Load rate also has no effect on the loading characteristics at this temperature. As an example of the increased rigidity at -100°F, a 25% deflection requires approximately 9,500 psi. It is significant that the material can withstand loads of this magnitude without failure and display resilience despite being about 40°F below the glass transition temperature. As mentioned earlier, the correlation between sealing capability and glass transition temperature has not been established.

Cryogenic sealing tests of AF-E-411 have not been conducted although a similar material (AF-E-71-2) has successfully sealed at temperatures as low as -423°F (Ref. 90).

Figure 4.2.20 shows hysteresis data for up to 100 cycles to 20% deflection at room temperature. As expected, repeated loadings, particularly the second, induces some softening of the rubber. At a nominal 20% deflection, the second cycle decreases the required load about 4.6%. After 100 loading cycles the total hysteresis loss is about 8.6% of the original load.

Compression set data are reported in Table 4.2-17 for tests conducted in air at various times, temperatures, and specimen conditions. Calculation Method B was used, as defined in Section 4.2.1. At temperatures up to +160°F, AF-E-411 has outstanding resistance to compression set. As the temperature increases, the compression set is increased appreciably as shown by the 300°F/720 hour data. At least two conditions of these tests are "worst case". First, the samples were 0-rings tested in air, AF-E-411's most prominent degradation agent. Second, the test specimens were not constrained in a seal gland but were free to expand. (This is the standard 0-ring compression set test method.) More air can circulate freely in an unconstrained geometry and the ring can set freely instead of encountering a restraining wall such as would be the case in an 0-ring groove. All measurements were taken immediately after unclamping (within one minute).

Table 4.2-18 reports compression set data for AF-E-411 measured after being compressed in propellant. At ambient room temperature the compression set values (determined by Method A) are very low (e.g., after 2160 hours at 75°F in 50/50, the compression set of AF-E-411 was 8.2% immediately after release). At 200°F, the compression set value in non-swelling N_2H_4 increased to 17.8%. As the methyl group concentration of the propellant increases from N_2H_4 to 50/50 to MMH to UDMH, the measured compression set decreases in the order of 17.8% to 10% to 7% to 0. This appears to be due to swelling counteracting the apparent set of the rubber.

While elastomers by definition are expected to recover from deformations very quickly, the data in Figure 4.2.21 show that additional recovery occurs in AF-E-411 for up to 24 hours after unclamping, although the majority of recovery occurs within one hour. Figure 4.2.22 illustrates the appearance of an 0-ring after elevated temperature exposure in 50/50. Little permanent set has occurred and no physical degradation as noted by by the "flash" remaining. The flash was not removed to allow evaluation of the compatibility of this section.

Some thermal properties of AF-E-411 are reported in Table 4.2-19. The low temperature at which a stretched, frozen sample regains its "rubbery character" in the absence of imposed work is the TR-10 temperature. AF-E-411 is a true elastomer at least as low as -40°F. However, the compression deflection data in Figure 4.2.19 shows the material will seal at temperatures considerably below -40°F when work is applied.

Thermal Gravimetric Analysis (TGA) is a sweeping air environment with a heating rate of 3°C per minute shows AF-E-411 to lose 1% weight at 561°F and that the material does not lose 50% weight until 865°F. Physical loss of this material, then, should not occur below approximately 560°F, although property degradation does occur.

In Table 4.2-20 the permeability constants of AF-E-411 with respect to helium, UDMH and hydrazine are shown. Permeability rates are shown in Figure 4.2.23.

Coefficient of Friction and Abrasion

The same techniques as used for AF-E-124D were used in obtaining coefficient of friction and wear data (see Section 4.2.1). The coefficient of friction data are shown in Figures 4.2.24 and 4.2.25 for 50/50, air, and varied temperatures as a function of surface speed.

Figure 4.2.26 gives wear as a function of the number of cycles at various loads. Although less wear occurs than with AF-E-124D at equivalent loads, wear occurs at a much higher rate than with Teflon.

Miscellaneous Properties

Other parameters determined during this program include specific gravity, and effect of vacuum exposure. These parameters are summarized in Table 4.2-21. The vacuum exposure results are essentially the same as for AF-E-124D. No appreciable change in weight or dimensions was noted as a result of this vacuum exposure. The vacuum level was 1.5×10^{-6} torr for a period of 168 hours.

Figure 4.2.27 shows the effect on seat stress of varying poppet surface finishes. The essentially constant stress is typical of an elastomeric seal. Within the range of measurement accuracy and test repeatability these data are essentially a constant value. These data are however primarily for information and comparison rather than design data. These tests were run (as described in Section 3.4) with a test fixture valve using a flat seal of approximately 1 inch diameter. These tests as well as the ones described in the AF-E-124D section were intended as demonstration and verification tests for the various materials. Zero leakage as defined for these tests was interpreted as no gaseous nitrogen leakage when measured by the water displacement method described in Section 3.4. Figure 4.2.28 shows the effect of exposure in 50/50 on required seat stress. Although the points vary, the stress appears relatively constant as a function of exposure time up to 24 hours. Figure 4.2.29 shows the seat stress required for sealing AF-E-411 preconditioned in 50/50 as a function of number of cycles. Up to the test maximum of 200 cycles only a small change was noted. Figure 4.2.30 shows the variation in required seat stress to seal at temperatures from -50 to +150°F. All values are threshold sealing stresses as obtained with the test configuration described.

	Temperature				
Parameter	-100°F	+75°F	+160°F	+300°F	
T _b , psi	7000	2300	1650	1100	
M ₁₀₀ , psi		1350		860	
E _b , %	45	170	280	125	
Tear, pli	1125	250	90	45	
Shore A		89			

Table 4.2-13

Effect of Air Aging on Mechanical Properties of AF-E-411

(Tested at Room Ambient Temperature

Hours Aged	Temperature °F	Property	Value (% Retention)
100	+300°F	T _b , psi	36%
		E _b , %	33%
		Shore A	+4
		ſear, pli	77%

AF-E-411 Mechanical Properties After Propellant Exposure (% Retention of Initial Properties) Table 4.2-14

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Parameter Control N ₂ H ₄ (1)	Control	N2H4 (1)		(L)HWON	50/50(1)	$\text{MMH}^{(1)} \text{UDMH}^{(1)} \text{50/50}^{(1)} \text{50/50}^{(2)} \text{50/50}^{(3)} \text{50/50}^{(4)} \text{50/50}^{(5)} \text{N}_{2}\text{H}_{4}^{(6)} \text{50/50}^{(7)}$	50/50(3)	50/50 ⁽⁴⁾	50/50(5)	N2H4 (6)	50/50(7)
M ₁₀₉ , psi 1650	1650	121%	121%	125%	121%	104%	100%	109%	112%	130%	38%
T _b , psi	2200	2111	102%	118%	107%	111%	108%	107%	105%	109%	100%
E _b , %	150	3 06	83%	1001	87%	2001	97%	1007	100%	93%	129%
Shore A ⁽⁸⁾	88	7	+2	-	+2	-5	+	0	+2	-	÷
Wt.Cng.,											
Ç G		; ;	::	1 1	11	+3.3	+3.8	4.0	+1.0 +1.0	!!	
Dim.Chg.% a) b)		1 1	! !	1 1	11	+2.9 0	+1.8 +0.6		1.9		; ;

(1) 7 Days @ R.T.* and Air Dry 7 Days at R.T.
(2) 3 Months @ R.T. and Air Dry 7 Days at R.T.
(3) 6 Months @ R.T. and Air Dry 7 Days at R.T.
(4) 9 Months @ R.T. and Air Dry 7 Days at R.T.
(5) 11 Months @ R.T. and Air Dry 7 Days at R.T.
(6) 100 Hours @ 16C°F and Air Dry 7 Days at R.T.
(7) 7 Days @ 200°F and Air Dry 7 Days at R.T.
(8) Shore change is change in Share Hardness Units from initial
(8) Immediately upon removal frc propellant
(8) After air dry for indicated time

-0.6400C840

(*R.T. = Room temperature = 70 - 75°F)

Table 4.2-15. Effect of AF-E-411 on Propellants

Exposure Time, Hours	Exposure Temperature °F	Propellant	Pressure Rise (above control) (psi	NVR
100	160	N ₂ H ₄	2.0	27.8
72	200	50/50	1.9	N/A

Table 4.2-16

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Mechanical Properties of AF-E-411 After Cleaning Solvent Exposure (14 Days at Ambient Temperature and Air Dry for 7 Days - % Change

	rluid				
Property	Freon TF	lrichlorethylene	Distilled Water	Isopropyl Alcohol	
T _b , psi	+10%	+13%	+18%	+ 9%	
M ₁₀₀ , psi	+18%	+ 18%	+15%	+21%	
E _b , %	- 7%	- 7%	-15%	- 7%	
Swe'', %	+11%	+ 24%	0	+ 1%	
Weight Change, %	+70%	+158%	, 0	+ 6%	
Shore A	+ 1	+ 1	+ 3	+ 1	

Table 4.2-17. Compression Set in Air of AF-E-411

Temperature °F	Time (Kours)	Specimen Configuration	% Initial Compression	% Compression Set
75°F	168	0-Rings	25	9.5
75°F	720	0-Rings	25	22.5
160°	96	0-Rings	25	14.0
160°	1024	0-Rings	25	31.5
200°	22	Buttons	25	17.0
300°	720	0-Rings	25	86.0

Table 4.2-18

AF-E-411 Compression Set in Propellants
(O-Ring Test Specimens)

Immediately Upon Release

Temperature °F	Time	Configuration	% Initia ¹ Compression	Propellant		ression et
					Method A	Method B
75°	2 months	0-Rings	25	50/50	9.5	33.3
75°	5 months	0-Rings	30	50/50	10.0	34.5
75°	3 months	0-Rings	30	50/50	8.2	28.6
75°	9 months	0-Rings	27	50/50	9.8	34.5
200°	22 hours	0-Rings	25	N ₂ H ₄	17.8	63.0
200°	22 hours	0-Rings	25	UDMH	0	o
200°	22 hours	0-Rings	25	MMH	6.0	22.0
200°	22 hours	0-Rings	30	N ₂ H ₄	17.8	64.0
200°	22 hours	0-Rings	30	50/50	10.0	35.2
200°	22 hours	0-Rings	30	UDMH	2.0	7.0

Method A: % Compression Set = $\frac{t_i - t_f}{t_i} \times 100$

Method B: % Compression Set = $\frac{t_1 - t_f}{t_1 - t_c} \times 100$

Where: t_i = initial thickness (0.1250* nominal)

 t_a = final thickness

t_C = thickness of compressed sample

Table 4.2-19
Thermal Properties of AF-E-411

Parameter	Property Value	
TGA (1) 1% Weight Loss 50% Weight Loss	561°F 865°F	(1) Temperature at which indicated weight loss occurs.
TR-10 (2)	-40°F	(2) Temperature at which elastomeric properties
Coefficient of Linear Thermal Expansion (1N/1N°F x 10 ⁻⁶)		reappear after being frozen at lower tempera ture (ASTM D-1329-60)
-100°F to -30°F - 30°F to +90°F + 90°F to 270°F	44.3 80.5 68.6	

Tabl∈ 4.2-20

	Helium, 70°F	UDMH, 70°F	N ₂ H ₄ , 70°F
	1 Atmosphere	l Atmosphere	1 Atmosphere
$p = \frac{(scc) (cm)}{(cm^2) (sec) (cm Hg)}$	25.8 x 10 ⁻¹⁷	1.7 x 10 ⁻¹⁰	1.5 × 10 ⁻¹⁶

Table 4.2-21
Miscellaneous Properties of AF-E-411

Property	Value	
Specific Gravity g/cc	1.03	
Vacuum Effects 168 hours at 1.5 x 10 ⁻⁶ torr		
% Weight Change	+1.4% max *	*Indicates no change
% Dimension Change	+0.3% max.*	within test accuracy

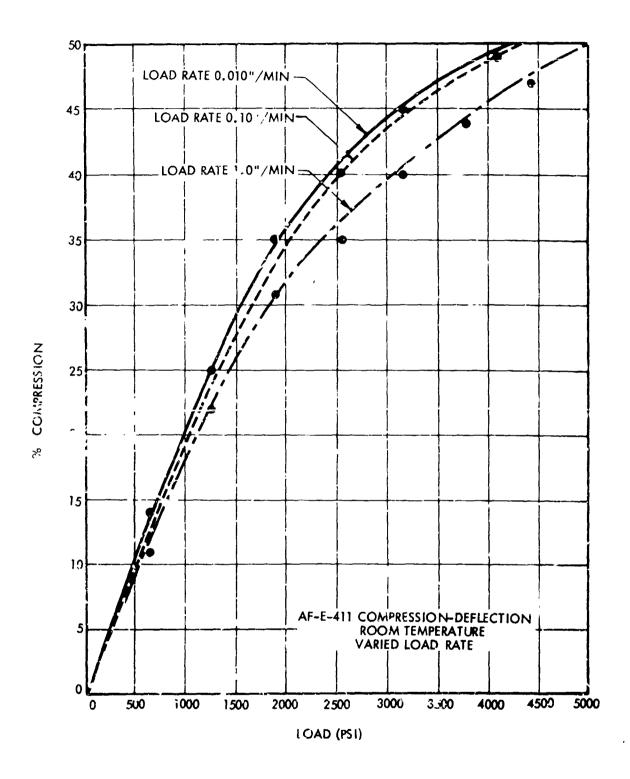
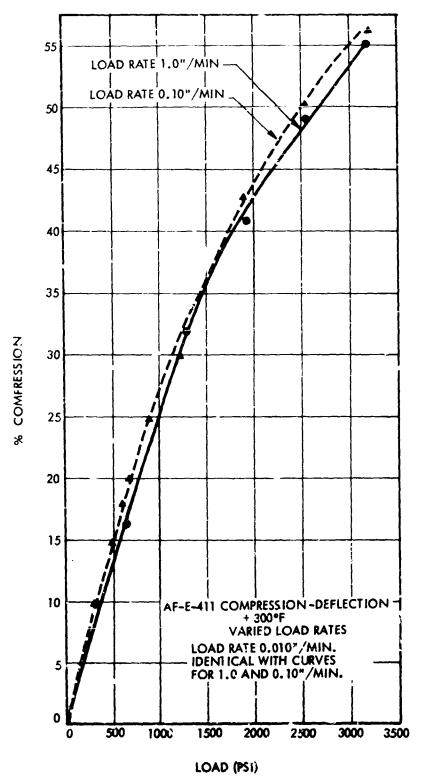


Figure 4.2.17 AF-E-411 Compression-Deflection Room Temperature, Varied Load Rate



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Figure 4.2.18 AF-E-4il Compression-Deflection +300°F - Varied Load Rates

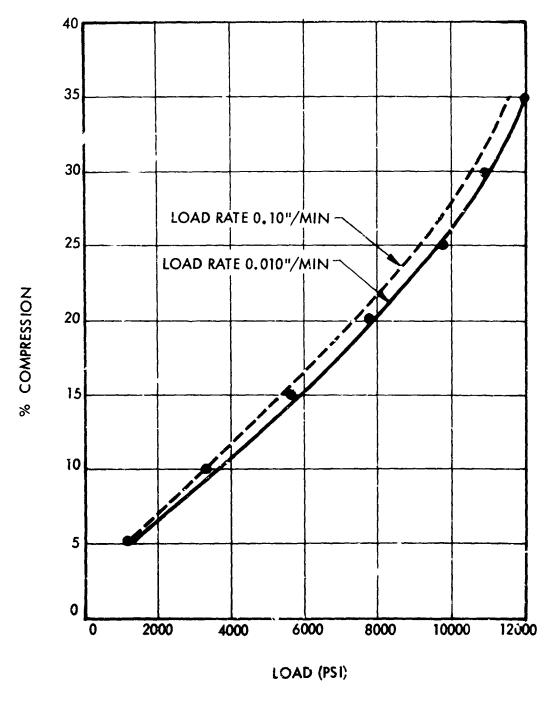


Figure 4.2.19 AF-E-411 Compression Deflection at -100°F Varied Load Rates

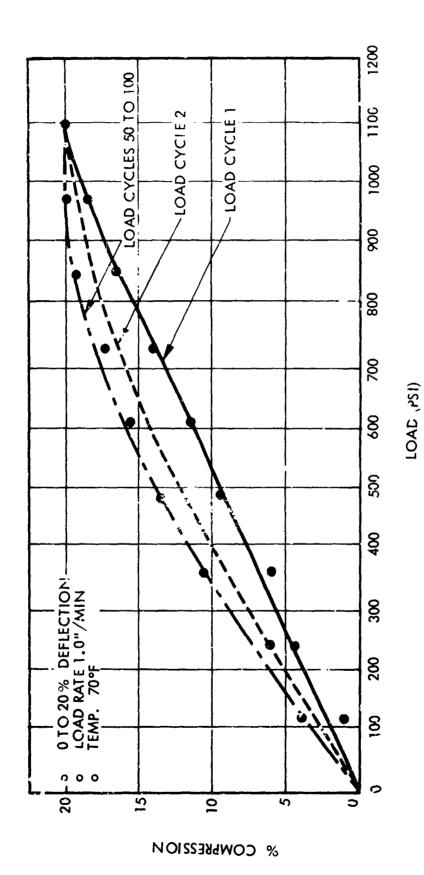


Figure 4.2.20 AF-E-411 Compression-Deflection Hysteresis

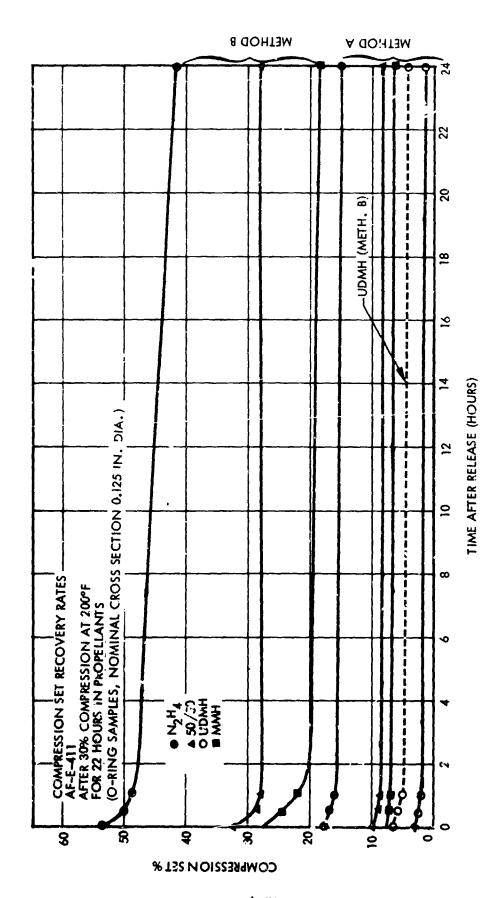


Figure 4.2.21 AF-E-411 Compression Set Recovery Rates

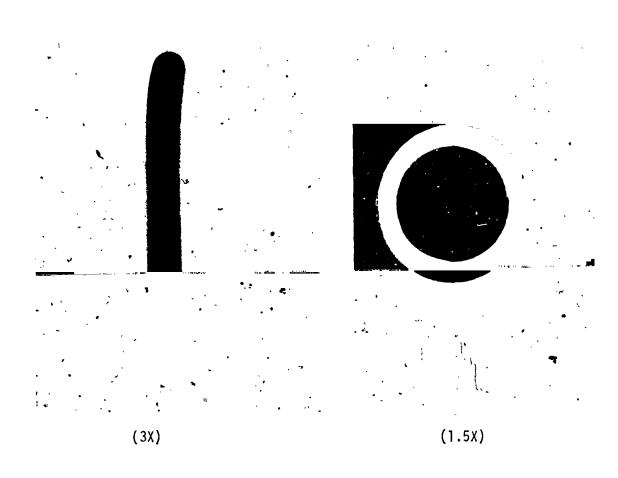


Figure 4.2.22 AF-E-411 O-Ring After 200°F/22 Hours
Exposure to 50/50 - 30% Compression
(Note: Original "flash" remains on I.D. and O.D.)

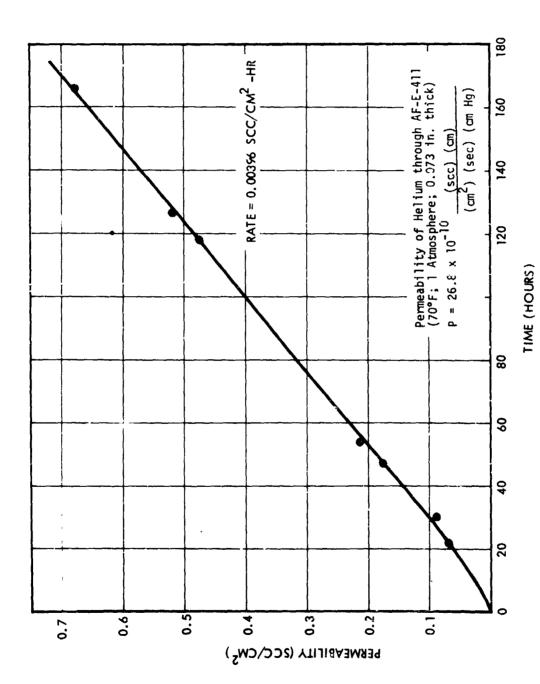


Figure 4.2.23 Permeability of Helium Through AF-E-411 (70°F; 1 Atmosphere; 0.073" Thick)

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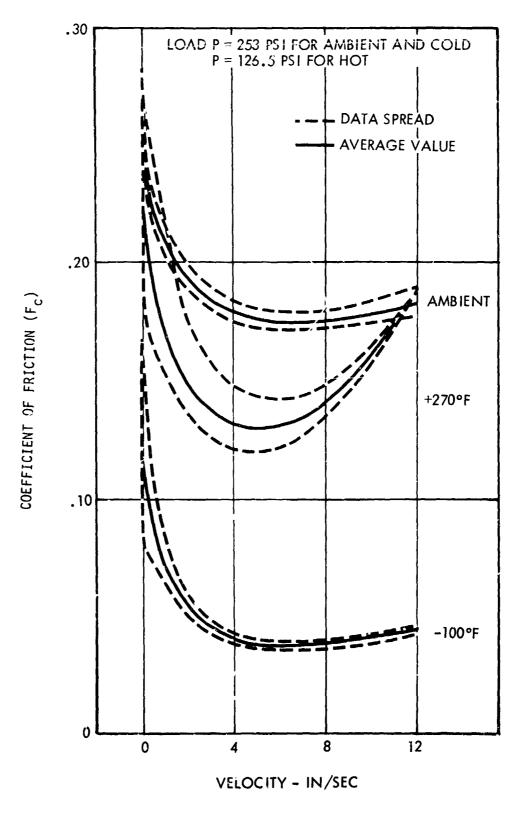


Figure 4.2.24 AF-E-411 Coefficients of Friction in Air

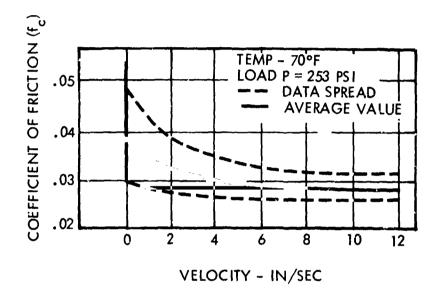
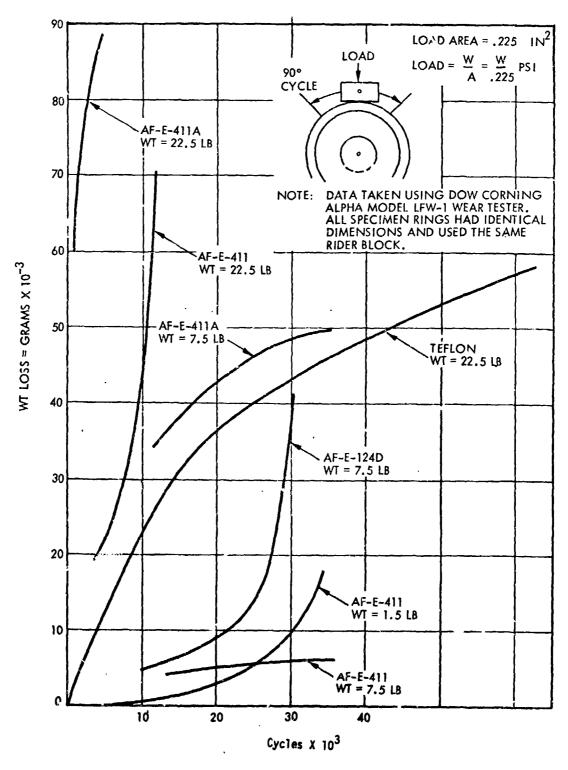


Figure 4.2.25 AF-E-411 Coefficient of Friction in 50/50



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Figure 4.2.26 Wear Comparison of Teflon & AF-E-411, AF-E-411A & AF-E-124U

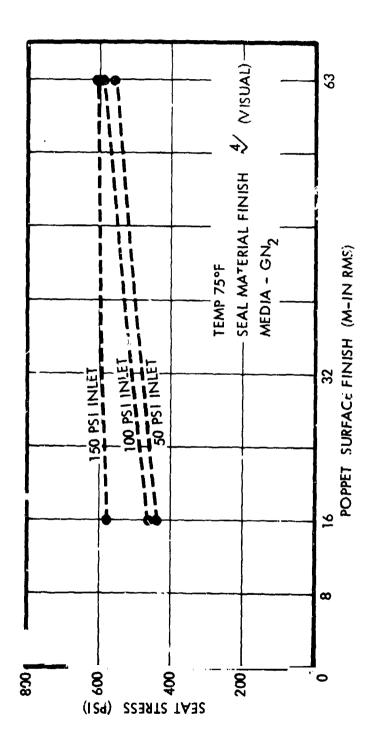


Figure 4.2.27 AF-E-411 Seat Stress to Obtain Zero Leakage* As a Function of Poppet Surface Finish (*Zero Leakage - No GN₂ Leakage Measured By Water Displacement)

SFAL FINISH 4 (VISUAL) INLET PRESSURE 100 PS1 POPPET FINISH TEMP 75°F 24 1 20 EXPOSURE TIME (HOURS) AF-E-411 50/50 Ar-E-124D 50/50-4F. E-1240 - N204 6705 (129) SE319TZ TA32 \$ \$ \$ \$ 200 8

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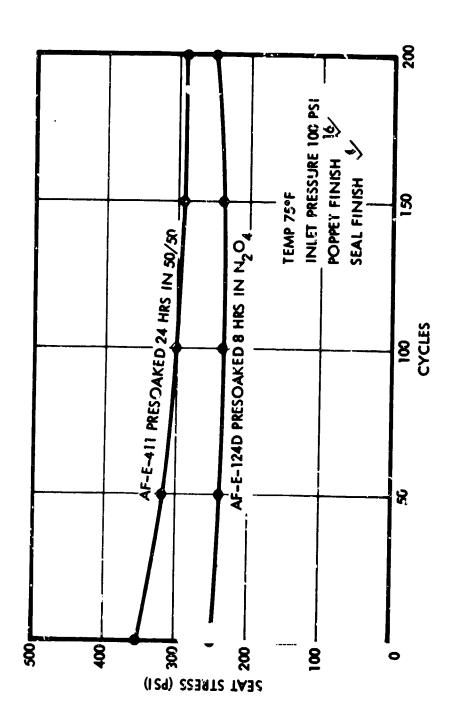
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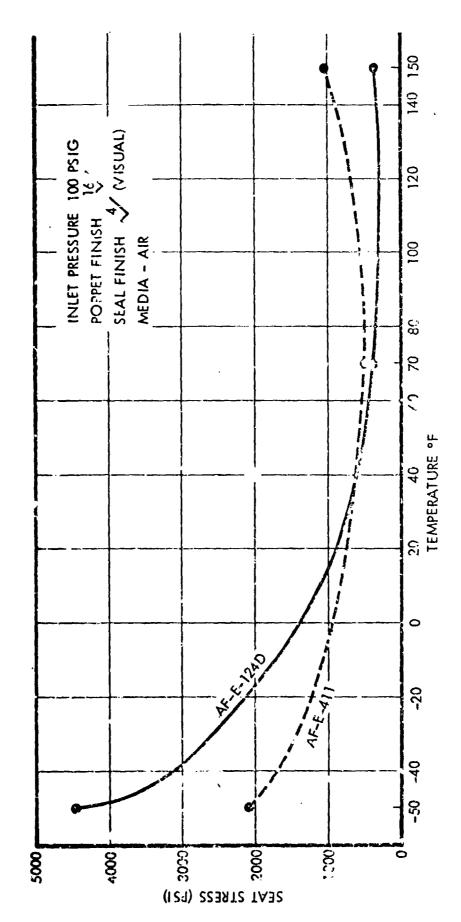
Figure 4.2.28 Seat Stress to Obtain Zero Leakage* As a Function of Propellant Exposure Time

(*Zero Leakage - No GN₂ Leakage Measured by Waver Displacement)

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(*Zero Leakage - No GN₂ Leakage Measured by Water Displacement Method) Seat Stress to Obtain Zero Leakage* As a Function of Number of Cycles Figure 4.2.29



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Figure 4.2.30 Seat Stress to Obtain Zero Lozkoge* As a Function of Temper: Lure

(*Zero Lerkage - No GN₂ Leakage Measured by water Lispincement)

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4.2.3 AF-E-411A

This material is essentially AF-E-4ll with an antioxidant (Age-Rite Resin D) added to increase elevated temperature thermal stability without sacrificing propellant compatibility.

Mechanical properties of AF-E-411A are reported in Table 4.2-22 over the range of temperatures investigated. Addition of the antioxidant slightly improves ambient room temperature properties, but decreases properties while at -100° F and $+300^{\circ}$ F.

Table 4.1-23 reports air aging data which show the expected advantage for AF-E-211A over --E-411. Even after 24 hours exposure to 400°F air, the modificul material retains elastomeric characteristics. At 300°F, AF-E-411 material is seriously degraded after 100 hours, while AF-E-411A retains most properties. The major advantage of the antioxidant is the retention of room temperature properties after exposure to elevated temperatures.

After exposure to fuels, AF-E-411A (Table 4.2-24) is somewhat inferior to the AF-E-411 base material in terms of retained mechanical properties. AF-E-411A apparently has a lower crosslink density and exhibits greater swell in propellant. Swelling dilutes the mechanical properties and apparent property degradation is probably the result of swelling in propellant.

Table 4.2-25 shows that the addition of antioxidant to AF-E-411 does not have a negative effect upon hydrazine compatibility. The lower NVR may result from less low molecular weight oils being generated in the presence of antioxidant during the peroxide cure reaction. The same cleaning solvent effects occurred as with AF-E-411 as shown in Table 4.2-26. Isopropyl alcohol is the recommended cleaning fluid.

Compression vs deflection curves at room temperature are shown in Figure 4.2.31 at various loading rates. Over two decades of loading rate the curves are within about $\pm 10\%$ of each other. The stress to obtain a given deflection is about the same as AF-E-411; to obtain 25% deflection, approximately 1100 psi is required.

At +300°F (Figure 4.2.32), AF-E-411A has about the same stiffness as at room temperature. This is probably because the polymer chains are more active and are on the average stretched somewhat tighter than at +75°F. At -100°F the material exhibits approximately the same properties as AF-E-411 with a load of 8,000 psi required for 25% deflection. At this temperature, 35% deflection was obtained without failure of material.

Figure 4.2.33 shows that up to 100 loading cycles AF-E-411A suffers some stress softening, but not a significant reduction.

Compression set in air for AF-E-411A (Table 4.2-27) is slightly poorer than AF-E-411. Both elevated temperature and room ambient temperature set is equal to or greater than AF-E-411. Apparently, where the surface area exposed to air is low, as in a confined or clamped configuration, the antioxidant offers no advantage and may actually increase set because of a lower inherent crosslink density.

Compression set in propellant (Table 4.2-28) also shows no advantage for adding the antioxidant to AF-E-411. Likewise, in recovery from deflection, as depicted in Figure 4.2.34 there is no advantage in adding antioxidant.

Retraction temperature (TR-10) is poorer for AF-E-411A than for the base stock. This may be due to the fact that less low molecular weight polymer fragments are generated during the peroxide cure and these oils are thereby less a factor in plasticizing the material. As shown in Table 4.2-29 the temperature at which 1% of the material is lose to sweeping air with a 3°C per minute heating rate, is improved by the antioxidant (from 561° to 649°F) indicating an increase in temperature capability before physical decomposition occurs.

Permeability of AF-E-411A to helium gas is shown in Table 4.2-30. The helium permeability coefficient was calculated from the data shown in Figure 4.2.35. This permeability value for helium of 19.3 x 10^{-10} is slightly lower than AF-E-411 (26.8 x 10^{-10}) and approximately 5 times lower than AF-E-124D. The permeability range would be expected to be similar to AF-E-411.

Miscellaneous properties are indicated in Table 4.2-31. As indicated for AF-E-411 no change occurred as a result of exposure to vacuum for 168 hours at 1.5×10^{-6} torr. Also shown in this table is the swell characteristic in 50/50 propellant. A small amount of swelling occurs after exposure but the material returns to the initial dimension after a drying period.

Coefficient of Friction and Abrasion

The same techniques as used for AF-E-124D were used in obtaining coefficients of friction and wear data (see Section 4.2.1). The coefficient of friction data are shown in Figures 4.2.36 and 4.2.37 for 50/50, air, and varied temperatures as a function of surface speed.

Figure 4.2.38 gives wear as a function of cycles at various loads, temperatures, and in comparison with Teflon, AF- E-124D and AF-E-411. The AF-E-411A material exhibits a much higher wear rate than the base AF-E-411 material. These data indicate that it should not be used in an application where sliding contact occurs to a high degree.

Table 4.2-22 Mechanical Properties of AF- E-411A

Parameter	Test Temperature	AF-E-411A	AF-E-411
T _b , psi	-100°F	4000	7000
E _b , %		50	45
Tear, pli		775	1125
M ₁₀₀ , ps1	+ 75°F	1350	1350
Tb, rsi		2500	2300
E _b , %		220	170
Tear, pli		225	250
Shore A		89	89
M ₁₀₀ , psi	+300°F	600	860
T _b , psi		825	1100
E _b , ≴		175	125
Tear, pli		85	45

Table 4.2-23
Effects of Air Aging on Mechanical Property Retention

Hours Aged	Temperature (°F)	Media	Froperty	AF-E-411A % Retained
100	+300°	Air	M ₁₀₀	107%
			Υ _b	92%
			Eb	82%
			Shore A	+1
24	+340°	Air	M ₇₀₀	135%
			T _b	98%
			Eb	77%
			Shore A	+2
24	+360°	Air	M ₁₀₀	154%
			Tb	105%
			Eb	63%
			Shore A	+1
24	+380°	Air	M ₁₀₀	
			T _b	82%
			Eb	51%
			Shore A	+2
24	+400°	Air	M ₁₀₀	
			T _b	57%
			Eb	34%
			Shore A	+3

AF-E-411A Mechanical Properties After Propellant Exposure (% Retention of Initial Prorenties) Table 4.2-24

Parameter Control N2H4	Control	N2F;4	HMH(1) n	(1)	50/50(1)	50/50(2)	50/50(3)	50/50(4)	$UDMH^{(1)}$ $50/50^{(1)}$ $50/50^{(2)}$ $50/50^{(3)}$ $50/50^{(4)}$ $50/50^{(5)}$ $N_2H_4^{(6)}$ $50/50^{(7)}$	N2H4 (6)	50/50:71
M ₁₀₀ , psi 1325	1325	121%	115%	128%	126%	328	84%	\$ 58	\$2 %	121%	86%
T _b , psi	2500	396	84%	%96	104%	87%	78%	85%	82%	85%	81 %
E _D , %	220	87%	82%	84%	87%	328	\$68	\$98	86%	71%	92%
Shore A ⁽⁸⁾	88	Ŧ	+5	0	7	7	7	+	Ŧ	, -	7
	_							_	•	-	

7 Days @ R.T. and Air Dry 7 Days at R.T.

6 Months @ R.T. and Air Dry 7 Days at R.T. 3 Months @ R.T. and Air Dry 7 Days at R.T. (2) (3)

9 Months @ R.T. and Air Dry 7 Days at R.T.

(4)

11 Months @ R.T. and Air Dry 7 Days at R.T. (2)

100 Hours @ 160°F and Air Dry 7 Days at R.T. (9)

7 Days @ 200°F and Air Dry 7 Days at R.T.

Shore Hardness change is Shore Hardness Units. (2)

 $(*K.T. = Room Temperature = 70 - 75^{0}F)$

Table 4.2-25
Effect of AF-E-411A on Propellants

Time of Immersion, Hours	Maximum Temperature °F	Propellant	Parameter	Pressure Rise
100	160	N ₂ H ₄	Pressure (a)	1.1
72	200	50/50	Pressure (a)	-6.3

- (a) 32 cm² surface area of material; 25 ml MIL-SPEC propellant; 71 ml ullage; all-glass containers. Pressure reported in psi above the control reading.
- (b) Non-volatile residue in units of mg/25 ml. Control was 9.8 mg/25 ml.

Table 4.2-25

AF-E-411A Mechanical Properties After Cleaning Fluid Exposure (14 Days at Ambient Room Temperature and Air Dry for 7 Days)

Cleaning Fluid	Property	%Change from Initial Value
Freon TF	M ₁₀₀ , psi	+ 25%
	T _b , psi	- 4%
	E _b , %	- 9%
	Swelling, %	+ 9%
	Weight Change, %	+ 84%
	Shore A	+ 1
Trichlorethylene	M ₁₀₀ , psi	+ 21%
	T _t , psi	+ 8%
	E _b , %	- 18%
	Swelling, %	+ 22%
	Weight Change, %	+194%
	Shore A	- 2
Distilled Water	M ₁₀₀ , psi	+ 23%
	T _b , psi	- 4%
	E _b , %	- 7%
	Swelling, %	<u>+</u> 0
	Weight Change, %	<u>+</u> 0
	Shore A	+ 1
Isopropyl Alcohol	M ₁₀₀ , psi	+ 21%
	T _b , psi	- 4%
	E _b , %	- 13%
	Swelling, %	<u>+</u> 0
	Weight Change, %	+ 2%
	Shore A	+ 1

Table 4.2-27

AF 5-411 Compression Set in Air

(Various Temperatures and Times)

(Immediately Upon Release)

Temperature	Time (Hours)	Configuration	% Initial Compression	Compression Set
75°	168	0-Rings	25	23.4
75°	720	0-Rings	25	24.0
257°	336	Buttons	25	37.5
300°	72	Buttons	25	14.7
300°	720	0-Rings	25	100.0

% Compression Set =
$$\frac{t_i - t_f}{t_i - t_c}$$
 X 100

Table 4.2-28
AF-E-411A Compression Set in Propellants (Various Temperatures and Times)

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75°F 2 months 75°F 3 months 75°F 5 months		Compression		Method A	Method B
	O-Rings	25	20/20	10.0	35.0
	0-Rings	30	20/20	7.6	26.7
_	0-Rings	30	20/20	11.0	38.5
75°F 9 months	0-Rings	30	20/20	7.8	26.6
200°F 22 hours	0-Rings	25	N ₂ H ₄	17.0	59.5
200°F 22 hours	0-Rings	25	HWON	2.0	7.0
200°F 22 hours	0-Rings	25	HWH	0.6	31.5
200°F 22 hours	0-Rings	30	N ₂ H ₄	17.0	0. ევ
200°F 22 hours	0-Rings	30	50/50	11.0	39.5
200°F 22 hours	0-Rings	30	HWQN	2.0	7.0

Method A: % Compression Set =
$$\frac{t_1}{t_1}$$
 x 100 Method B: % Compression Set = $\frac{t_1}{t_1}$ - $\frac{t}{t_C}$ x 100

Where: t_i = initial thickness (0.1250 NOM) t_f = final thickness t_c = thickness of compressed sample

Table 4.2-29
Thermal Properties of AF-E-411A

Parameter	Propert; Value	Comments
TGA ⁽¹⁾ 1% Weight Loss 50% Weight Loss TR-10 ⁽²⁾	649 ⁰ F 836 ⁰ F -22 ⁰ F	(1) Temperature at which indicated weight loss occurs. (2) Temperature at which elastomeric properties reappear after being frozen at lower temp.
Coefficient of Linear Thermal Expansion (in/in x 10 ⁻⁶) -100 ⁰ F to -30 ⁰ F - 30 ⁰ F to +90 ⁰ F + 90 ⁰ F to +325 ⁰ F	42.2 80.5 69.1	

Table 4.2-30
Permeability Coefficient of AF-E-4!1A

Conditions	p*
Helium, 70°F, 1 Atmosphere	19.3 x 10 ⁻¹⁹
*p = <u>(scc) (cm)</u> (cm ²) (sec) (cm H	g)

Table 4.2-31
Miscellaneous Properties of AF-E-411A

Property	Value
Specific Gravity, (grams/cc)	1.03
Vacuum Effects - 168 Hours at 1.5 x 10 ⁻⁶ torr % Weight Change % Dimension Change	+0.8% max. (a +0.4% max. (a
Swell in 50/50 inches (linear)	
(b)	
(1)	2.5%
(2)	~ 0

- (a) Approximately zero within test accuracy
- (b) 1 As soon as removed from $prop_{\ell}$ llant
 - 2 After 7 day outgassing per 10d

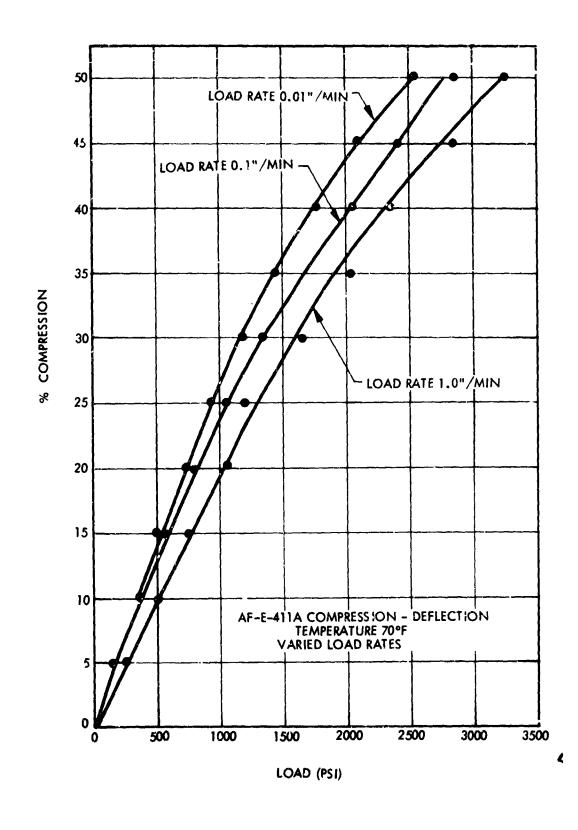


Figure 4.2.31 AF-E-411A Compression-Deflection Temperature 70°F - Varied Load Rates

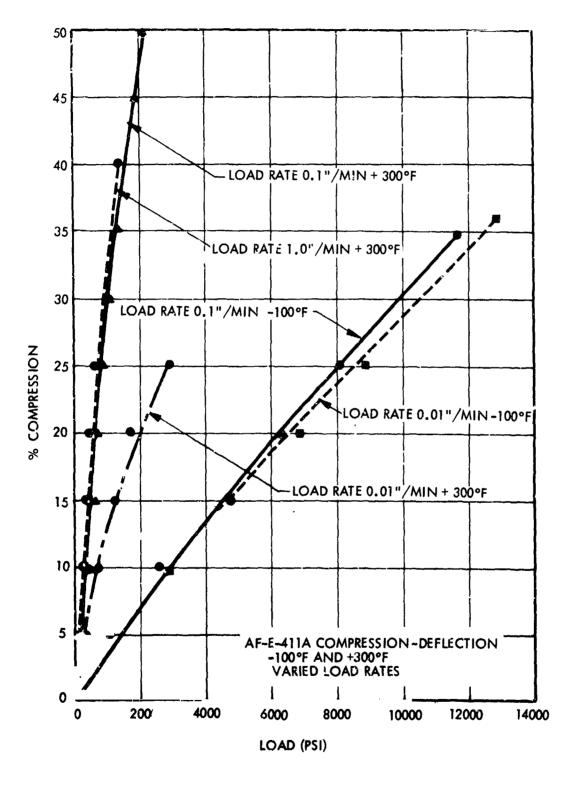
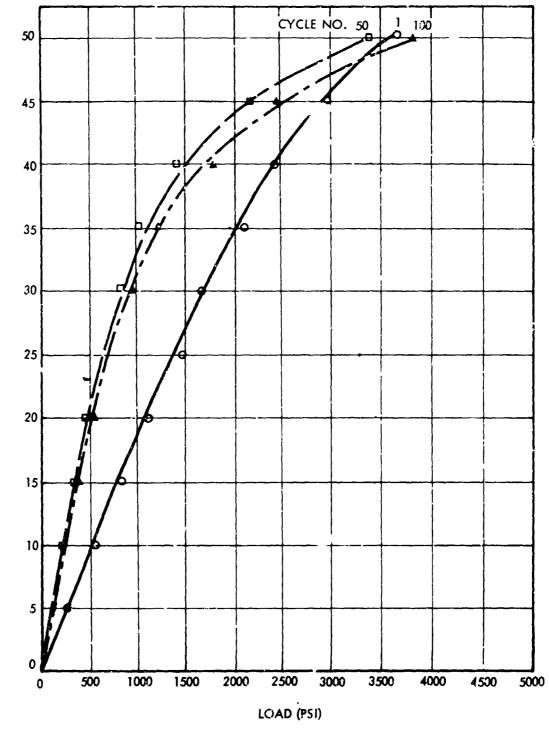


Figure 4.2.32 AF-E-411A Compression-Deflection -100°F and +300°F Varied Load Rates



% COMPRESSION

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Figure 4.2-33. AF-E-411A Compression-Deflection Hysteresis and Repeatability (at 70°F)

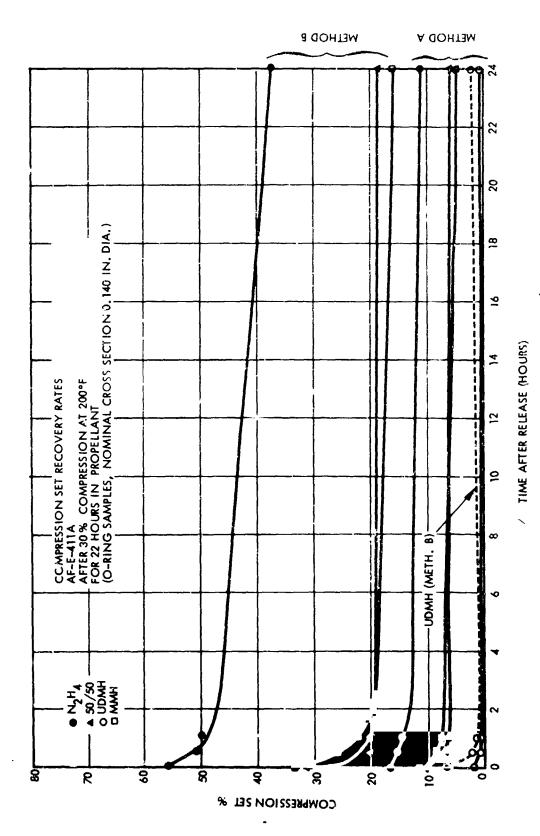
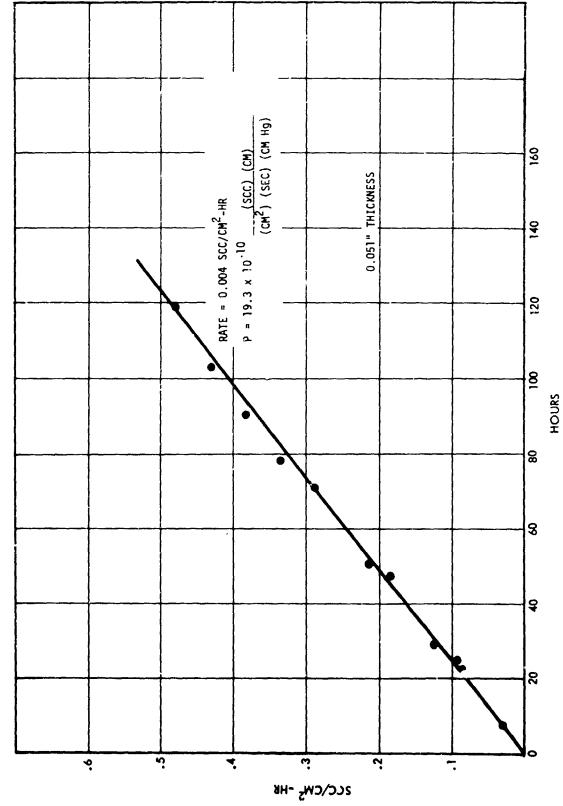


Figure 4.2.34 AF-E-411 Compression Set Recovery Rates



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Figure 4.2.35 Helium Permeability Through AF-E-411A

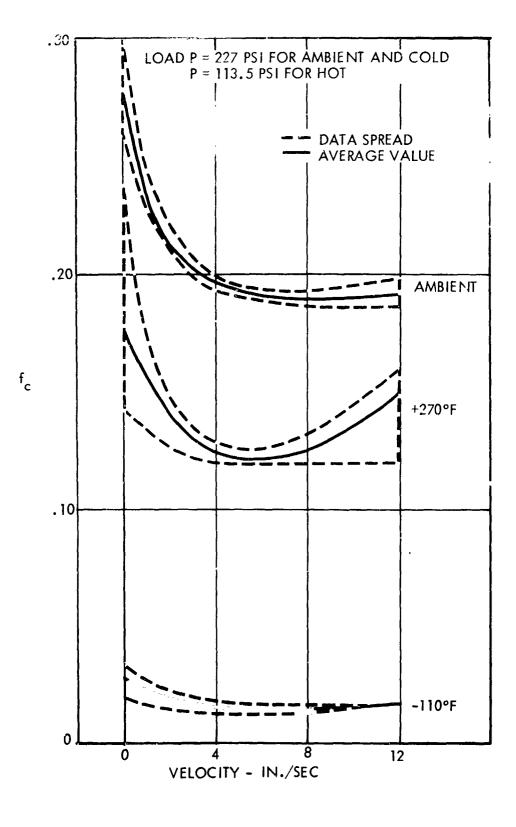


Figure 4.2-36. AF-E-411A Coefficients of Friction in Air

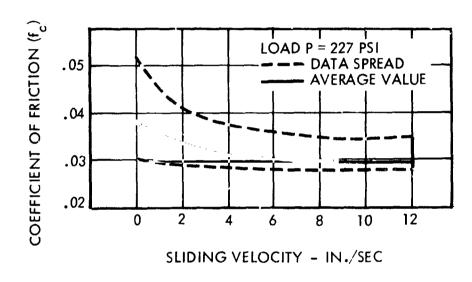


Figure 4.2.37 AF-E-411A Coefficient of Friction in 50/50

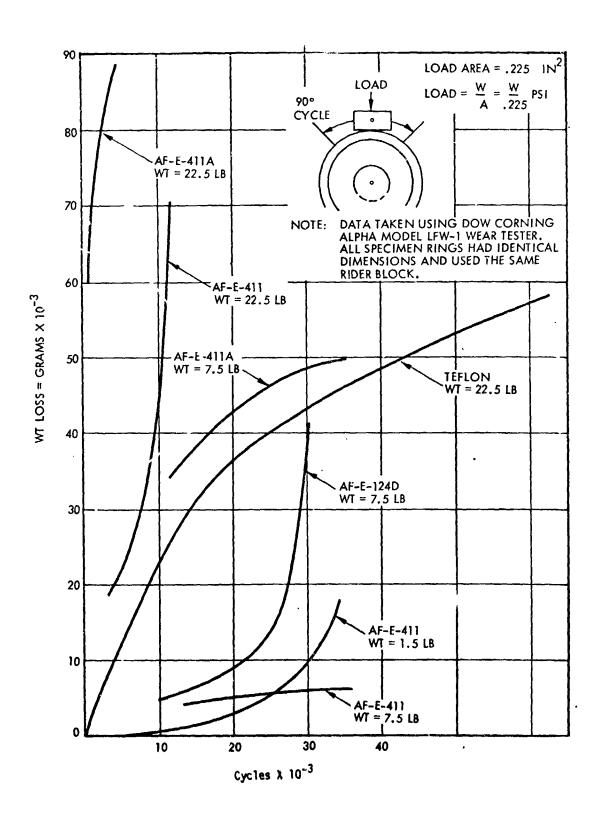


Figure 4.2.38 Wear Comparison of Teflon & AF-E-411, AF-E-411A, & AF-E-124D

4.3 PROPERTIES OF OTHER SEAL MATERIALS IN COMPANUSE

4.3 Properties of Other Seal Materials In Common Use

Seal materials commonly used in propulsion system sealing applications were identified in the state-of-the art investigation. This investigation surveyed literature, manufacturers and government sources to identify and document seal application and materials. Tables 4.3-1, 4.3-2, and 4.3-3 are taken from Ref. 86 to indicate typical applications and materials used in bipropellant and monopropellant applications. In Table 4.3-3 more advanced or current systems are listed both bipropellant and monopropellant, to indicate current seal usage. These tables are obviously not intended to summarize all applications, but are considered representative of types of seals and materials.

This section is designed to provide information on those non-metallic seal materials most commonly used or most applicable. As seen from these tables these materials are: Teflon TFE and FEF, Filled Teflon, Kel-F and EPR. In addition, materials used in system functions not directly in contact with propellant are included. These materials are: Kynar, Vespel and Viton. Carboxynitroso rubber (CNR) has been used for oxidizer seal applications in Apollo and is also included.

This section also includes a brief description of the seal materials, with sufficient properties data to allow the seal designer to conduct a preliminary evaluation of the material. Applicable references are noted to provide more information where desired.

Table 4.3-1. Representative Monopropellant Hydrazine System Valves

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					Seal	_	Seat	t	Leak	Leak Rate Specif.	ن		
Vehicle or Stage	Engine or System Supplier	Valve Type/ Function	Pressure	Flow Rate 15/sec	Material	Config	Material	Config	lest Fluid	Test Pressur psig	Allow. Rate SCCH	Cycle Life Spec.	Valve Manufacturer
Intelsat III	2	Poppet- Thruster	009	0.015	EPR	Flat	304.	Flat	E	009	0.5	000,09	Fairchild Industries
Planeer F&G	2	Poppet- Thruster	<u>8</u>	ı	EPR	Flat	304L	Flat	GN2	009	0.5	100,000	Allen Design Inc.
38	7	Poppet- Teruster	<u>8</u>	0.015	EPR	Flat	304	Flat	GN2	069	0.5	000.09	Fairchild, Allen Design, Parker-Hannifin
	136	Poppet- Thruster	22	1	EPR	Flat	304	Flat	GN2	50	0.5	1 × 10 ⁶	Allen Design Inc.
11 SOS	T. N.	Poppet- Thruster	009	0.015	EP.	Flat	304L	Flat	GN2	009	0.5	000.09	Fairchild Industries, Parker-Hennifin
Intelsat IV	thegres	Poppet Torque Motor Thruster	% %	1	Tungs ten Carbide	Flat	Tungsten Carbide	Flat	GN2	l	5.0	i	Hydraulic Research Corp.
Intelsat IV	Hughes	Poppet Isolation	900	!	Teflon	Ring	300 CRES	Confcal	GN2	300	2.0	000	Carleton Controls
ATS F&G	Rocket Research	Poppet- Thruster	9	0.00045	Teflon TFE	Flat	30¢L	Flat	GN2	90	8.	1 × 106	Parker-Hannifin
ERTS A	Rocket Research	Poppet- Thruster	400	0.0022	Teflon TrE	Flat	304L	Flat	GN2	004	9.0		Parker-Hannifin
P-95	Rocket Mesearch	Poppet- Thruster	30	0.012	Teflon TFE	Flat	304L	Flat	GN2	300	٥٠،	1 y 10 ⁵	Parker-Hannifin
P-95 (Alternate)	Rocket Research	Poppet- Thruster	300	0.012	Teflon TFE	Flat	304L	Flat	GN2	300	0.0	1 x 10 ⁵	Moog Inc.
Transtage	Rocket Research	Poppet- Thrus ter	400	711.0	Teflon TFE	Flat	17-7PH	Flat	GN2	360	5.0	72,000	Moog Inc.
RVE-8	Hamilton-Standard	Poppet- Thruster	522	1	Tungs ten Carbide	Flat	Tungs ten Carbide	Flat	3 5	210	5.0		Hydraulic Research Corp.
88	Rocket Research	Poppet- Thruster	Į	ı	EPT/HYSTL	1	1	1	1	1	1		Parker-Hannifin
										— <u>—</u>			
Solrad X	Hamilton-Standard	Poppet- Thruster	350	0.003	EPT/HYSTL	Flat	304	Flat	뢇	350	0.007	2 x 10 ⁶	Wright Components
Can. Tech. Sat	Hamilton-Standard	Poppet- Thruster	200	0.0015	EPT/HYSTL	Flat	304	Flat	GW2	700	2.0	1 x 106	Wright Components
HAT TAN	Rocket Research	Poppet- Thruster	350	0.022	Teflon	Flat	304L	Flat	S.	350	4.0	x 105	Parker-Hannifin
728	Rocket Research	Poppet- Thruster		1	Teflon	Flat	3041	Flat	1	1	1	1	Parker-Hannifin
								1		1			

Table 4.3-2. Representative Bipropellant System Valves

					1		Sea	,	Sec	t			Rate ec	
Vehicle or Stage & Subsystem	Engine & Supplier	Fluid	Valve Type	Valve Manufactu.er	Pressure psig	Flow Rate 16/sec	daterial	Config	Material	Config	Test Fluid	Test Press psig	Allow Rate SCCH	Cycle Life Spec
inuteman III PBPS Axial)	LR-123-NA-1 (Rocketdyne)	N204	Torquemotor Poppet	Moog -	247	0 678 0 424	Teflon TFE Teflon TFE	Flat Flat	17-7PH 17-7PH	Flat Flat	uN.	247	5 0 5 0	10,000
inutemen III PBPS RCS)	LR-125-8A-1 (LAC)	N204 1884	Torquemotor Poppet	Moog	247	069 055	Teflon TFE Teflon TFE	Flat Flat	17-7PH 17-7PH	Fiat Flat	GH2	247	5 0 5 0	10,000
eriner Hers 71	RS-2101 (Rocketdyne)	N ₂ O ₄	Torquemotor Poppet	Hoog	300	0.68	Teflon TFE Teflon TFE	Flat Flat	17-7PH 17-7PH	Flat Flat	GH2	300 300	5 D 5 O	
pollo SM RCS	R-4D (Kerquardt)	N204 A-50	Salenoid Poppet	Marquardt	185	0.24 0.12	Teflon TFE	Beveled Disc	Stellite No 6	Comical Poppet	CN5	200	5.0	
pollo LM RCS	R-4D-2 (Marquardt)	N204 A-50	Salenoid Poppet	Marquardt	195	0.24	Teflon TFE	Bevaled Disc	Stellite No 6	Confcal Poppet	GN2	200	5.)	
pollo ystem isolation	N/A	N204 A-50	Latch Solenoid Poppet	Parker	250	1.0	Teflon TFE	Flat	304L	Flat	He	250	10 0	1,00
pollo LM Ascent	(Pockatdyne)	N,04 A-50	Ball(Quad)	BAC	205	7.0	Teflon TFE	Ring	302	Ball				
pollo LM Descent	(TRM)	N204 A-50	Ball (Quad)	Whittaker	231	19.9 12.5	Teflon TFE	Lipseal	17-4PH	Partial Ball	GN2	250	0 1 SC14	
pollo LM Prevalve	N/A	A-50	Jolenoid Poppet	Valcor	280	12.5	EPR	Retained 0-Ring	304L	Conical	GN2	250	15.0	
pollo SM	A210-137 (AGC)	N204 A-50	3a11 (Quad)	A GC	2 25	38 24	Teflon Glass- filled	Lipseal	17- 4 PH	Ball	GH ₂	150	200 (600/ valve)	100
unar Orbitar	R-40 (Marquardt)	:: ₂ 04 A-50	Poppet	Marquardt	190 190	0.24 0.12	Teflon TFE	Beveled Disc	Stellite No. 6	Conical Poppet	GN2	200	50	
MPS	URSA-100R (T#≌)	NZO4	Torquemotor Poppet	Moog	300	0.182	Teflon TFE	Flat	17-7PH	Flat	GN2	235	5.0	25,0
genä	LR-R1-BA (BAC)	IRPM, UDM	Poppet Poppet	BAC BAC	1 000 900	35.0 15.0	Teflon EPR	Conicel Conicel	304L 304L	Conical Conical	GN2 GN2	1000 900	60 60	50 50
gena - Propellant solation	R/A	IRFNA UDM	Gate	Whitcaker	100		Teflon	Cartridge	CPES	Flat Belleville	GN2	100	6x106	5,00
itan Transtage	AJ 10-138 (Aerojet)	N204 4-50	Poppet	AGC	250	22.6 11.3	Teflon	Lipscal	CRES Alum		GH ₂	250	30	5,0
ftan Stage 2	(Aarojet)	N204 A-50	Butterfly	AGC AGC	1113 1195	1000 500	rel-F Nylon	Lipseal Lipseal	17-4PH 17-4PH	Butterfly Butterfly	GN ₂	1100	1900	10
itan Stage I	LR87-A5 (AGC)	N204 A-50	Butterfly	AGC	1057 1358	440 220	Kel-F Nylon	Lipseal Lipseal	17-4PH 17-4Ph	Butterfly Butterfly	GN ₂	1100	1900	10
itan Transtage CS	SE-9 (Rocketdyne)	M204 A-50	Poppet	Rocketdyne	305		·		!				4	
ertni	SE-6 (Rocketdyne)	N204 N991	Poppet	Rocketdyne	295	.0467	Teflon FEP		Stellite No. 3					
emini DAMS	SE-7 (Rocketdyne)	N204 N904	Poppe t	Rocketúyne	295	0,158 0,131								
pollo CM	SE-8 (Rocketdyne)	N204 HMH	Poppet	Rocketdyre	300	0.230 0.109	Teflon	Ring	CRES	Poppet	GH2	100	0.2	
aturn IVB RCS	700800 (TRu/)	H2O4 HHH	Solenoid Poppet	Valcor	195	0.312	Teflon	Flat	3041	FlatPoppat	Liq	275	0.021	10,400
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Rocketdyne - Rocketdyne Division, Rockeel International Corp. Marquardt - Marquardt Corp TRN - TRN Systems Noog - Moog Inconporated Parker - Parker Hannifin Corp.

Table 4.3-3. Valves in Advanced or Non-Operational System (Bipropellant and Monopropellant)

1

Valve Type	Application	Flufd	Valve Supplier	System Supplier	Pressure	Nowinal Flor Pare	3		3	_		Leak Rate Spec.	Spec.	Cycle
				antena a	Ps1g	T	Material	config.	Material	Con719.	Test Fluid P	Test d Pressure Psiq	Allowable Rate SCCH	Life Req'd,
forgumo tor Poppet	ISPS Engine	M ₂ O ₄	Moog	125	300	0.270	Teflon TFE	Flat	304.	Flat	2 8	25	5.0	25,000
utterfly	2 <u>2 2 3</u>	#20#	Post-Seal Inc.	170k	300	19.6	Teflon AF-E-124D Teflon	Rtng	316	Butterfly	ž²	8	10.0	500 200
wterfly	Titan Prevalve	0 × 4	Martin-Marietta	Martin-Marietta	\$8	4,6,7" Valve Diameter	Teflon	Ring	CRES	Butterfly	ž	85	3 x 10 ⁻¹⁰	0001
														
Solenoid-poppet	AE Thruster Yelve	* _K ~ _N	Parker	. R.	99	0.015	EPT/HYSTL	Flat	304L	Flat	3 2	99	0.5	000'09
Solenoid-pappet	AE Tank S/0	¥ ₂ ¥4	Moog	101	6	0.02	Teflon	nat	304L	Flat	₹ *	009	0	000,001
Solenoid-pappet	P-50 Thruster Valve	* 12 N	Parker	Hamilton-Standard	255	0.050	Teflon TřE	Flat	304L	Flat	5 ~	255	0.3	1.5 × 10°
Solemoid-goppet	P-50 Thruster Valve	*24¢	Parker	Hamilton-Standard	255	0.0025	Teflon TFE	Flat	3 0€	Flat	<u> </u>	255	5.0	15 x 10
Solenoid-pappet	Mariner '73 Thruster Valve	12H4	Narquardt	JP.	38		Teflon TFE	Beveled Disc	Stellite No.6	Cor 1cal porpet	5 °	88	5.0	
Solemoid-pappet	Mariner '75 Isolation	N2H4	Farker	JPL		(Line Size)	Teflon TFE	Flat	304L	Flat	5 ²	250	10.0	

4

4.3.1 Teflon TFE, FLP, and Filled

The most commonly used family of seal materials in storable propellant use today are those made of Teflon or Teflon compounds. Because of this, more information and discussion is provided on these materials than others.

4.3.1.1 Teflon TFE

Polytetrafluoroethylene (TFE or PTFE) is called Teflon in this report as a matter of convenience. Actually, "Teflon" is the registered trademark of the E. I. du Pont de Nemours and Co. (Inc.) and legally should be used only to specify their resin materials or the solids made from those resins. TFE is also marketed by several large chemical suppliers under their own trademarks.

The chemical formula of TFE is: $(CF_2CF_2)n$. Its monomer can be shown diagrammatically as:

Thousands of these monomers join together to form long chains of polytetrafluoroethylene:

(Some of the information in this section is also from Reference 49.)

Thick pieces of TFE are opaque and milk white in color. Thin pieces are translucent, and very thin films are almost transparent. Any coloring, other than the milk white, is due to foreign materials such as residual wetting agent, fillers, etc., which may have been added during processing, or to fluids which have permeated into the structure after processing. High purity TFE is now available from specialty houses who process the resins with great care to exclude trace impurities.

TFE is supplied by the manufacturer in the form of molding powder or aqueous dispersion. Processors form these raw materials into solid pieces by pressure molding (the powder) or dip coating (with the dispersion). The parts so formed can be strengthened by sintering to coalesce the particles. Postforming, by the application of pressure at temperatures approaching but below the transition temperature (620°F), can be used to slightly alter the shape of the sintered parts but radical reshaping is difficult. This difficulty stems from the fact that, above the transition temperature, TFE resins enter into a gel state (at 620°F) which limits the melt flow. Because of its unresponsiveness to heat forming methods, TFE is not considered to be a true thermoplastic. Seals generally are either molded from the powder or machined from stock.

The most useful property of TFE, for the design engineer, is its inertness. It is the most inert polymer known. Only molten alkali metals, fluorine, and certain halogenated compounds will react with it. De_pite its inertness TFE will be physically affected by some substances, including nitrogen tetroxide, which permeate into it and cause swelling; removal of the permeant will restore TFE to its original condition.

Another valuable property is the very low coefficient of friction of TFE. Because of this the surface has a slippery feeling even when chemically clean. Its lubricity is so outstanding that solid pieces of TFE or TFE-base materials are used as journal bearings, piston rings, etc., without additional lubricants. TTE also is applied by dip coating or spraying from an aerosol bomb to form thin, solid lubricating films on solid substrates.

Related to its low-friction characteristics is the difficulty in attaining a really secure bond between TFE and other materials using conventional techniques. Special processes are a necessary pre-requisite to

producing high quality structural join's. A moderate degree of success has been achieved in heat conding TFF to thin metal foils but such bonds usually fail when, for example, the TFE is saturated with nitrogen tetroxide.

Another characteristic of value is TFE's very high impact strength which resists fracture as when a poppet valve closes very rapidly.

The bulk properties of TFE can also be related to the spatial orientation of the polymer chains. In many regions the chains are in random orientation relative to each other; this portion of the film is said to be in the amorphous state. In the remaining regions, chains are arranged in a more or less orderly fashion which is described as the crystalline state; it is believed that the chains tend to orient themselves parallel to each other in the regions of crystallinity to form clusters much like bundles of rods. These two states are co-existent in all TFE material.

Crystalline regions and amorphous regions are interspersed so the bulk properties reflect an average response of both states. For example, the more ordered portions of the chains are less free to be reoriented by external forces than the randown parts so they contribute a degree of rigidity to the entire mass, and it follows that the rigidity becomes greater as the proportion of crystallinity to amorphous content increases. Other properties are also affected by this same proportion. To numerically express this proportion, the percentage of the total mass which is in the crystalline state is termed the crystallinity of the TFE, e.g., if 53 percent by weight of the total is crystalline, then the crystallinity is 53 percent.

Not all of the volume apparently occupied by the TFE is packed with polymer chains. Vacant spaces occur because TFE is not a completely uniform crystalline substance. The degree to which all of the space is occupied in a finished piece of TFE depends upon a number of factors including the size, shape and porosity of the particles in the resin and the processing the resin undergoes as it is formed into the solid film. By measuring the specific gravity of the finished material and comparing it with the theoretical specific gravity for voidless TFE of the same

crystallinity, the percentage of void space in the finished material can be calculated. Expressed as a percentage of the total volume, the answer is called the void content.

When TFE is being processed into a seal, the three factors of molecular weight, crystallinity, and void content should be adjusted within the available limits to develop a seal with the best properties for the intended use.

Tensile strength is affected by all three of the basic factors previously mentioned: average molecular weight, crystallinity, and void content. Tensile strength increases with increasing molecular weight. The molecular weight cannot be increased over that originally present in the resin but it can be decreased unless thermal degradation is avoided. Sintering temperatures above approximately $+734^{\circ}F$ may result in degradation by the successive removal of monomers CF_2CF_2 from the ends of the chains.

Tensile strength is best at low levels of crystallinity. Low crystallinity is achieved by quick quenching after sintering.

Low void content makes for good tensile strength. Void content is not very controllable since it is largely a function of the size, shape and porosity of the particles in the resin; careful control over the sintering temperature will, however, help to avoid unnecessary increases in void content.

It can be seen from the above that the tensile strength of a part produced from a given resin is maximized if the proper sintering temperature is carefully maintained and then the part rapidly quenched. This process results in a part with high average molecular weight, low crystallinity, and low void content.

While the average molecular weight is desirable in that it yields high tensile strengths, the elongation is less than maximum at this condition. Shorter TFE chains give better elongation. To a degree, the same dilemma is faced in the choice of crystallinity objectives because higher crystallinity, up to 85 percent, results in greater elongation as

opposed to the need for lower crystallinity for best tensile strength. A low void content gives both extra elongation and extra strength.

Optimizing TFE becomes even more complicated, however, when a low flexural modulus is desired. Average molecular weight does not seem to have much effect but crystallinity should be low, as with tensile strength, and void content should be high, contrary to the need for low void content to get high tensile strength and great elongation. To illustrate the sensitivity of flexural modulus to crystallinity, fast quenching to keep crystallinity low may yield a modulus as low as 55,000 psi while slow annealing may increase the crystallinity enough to raise the modulus to 90,000 psi.

Based on experience with TFE dilms, a high average molecular weight and low crystallinity are the best compromise to achieve durability. This seems to imply that tensile strength and flexural modulus are more important than elongation. This is corroborated somewhat by the fact that Du Pont reports that flex life is increased by adjusting the basic factors in the same way as required to increase tensile strength.

In some cases the demand for low permeability may create an overriding consideration in favor of low void content. Permeability to most
fluids used in rocket systems is strongly a function of the void content
— less void giving less permeation. To a certain degree permeability is
affected by the crystallinity (decreasing permeability with increasing
crystallinity) but average molecular weight seems to be insignificant in
connection with permeability.

It should be noted that void content may drastically affect other properties as well. Reference 59 reports that void content in TFE can reduce flex fatigue life by 1000 fold, increase permeability by 1000 fold, decrease Rockwell Hardness by 30 percent, decrease Tensile Impact Strength by 80 percent, decrease the Proportional Limit and yield stress both by 20 percent, decrease Ultimate and Tensile Strength by 50 percent, and decrease Ultimate Elongation by 30 percent. Hence, density measurements are recommended to ascertain the void content in order to predict the degradation in properties to be expected.

TFE density changes about 6 percent when the crystallinity varies from 47 to 84 percent. Actual density, P. is reduced in direct proportion to the void content:

$$\rho = \rho_0 \left(1 - \frac{V}{100}\right)$$
 where: ρ_0 = inherent density at zero void content V = void content as a percentage

Temperature also affects density. Thermal expansion/contraction data should be consulted to determine the magnitude of density changes versus temperature. TFE decreases in volume by about 4.3 percent when chilled from $\pm 68^{\circ}$ to $\pm 100^{\circ}$ F (Reference 85).

The calculated bulk compressibility of TFE is 2.07×10^5 psi when kept under load for 200 hours; and 1.57×10^5 psi when under load for one year. Poisson's ratio for TFE is 0.46.

TFE is slow to recover after being strained, recovering only 59 percent of its compressive strain when the reduced time is 1 (i.e., recovery time is equal to the time under load).

TFE apparently experiences elastic wear at rates which are approximately proportional to the modulus of elasticity and inversely proportional to the yield strain (Reference 54). Reference 30 suggests use of the PV relation to estimate the wear of Teflon:

TFE is among the most severely degraded of materials when exposed to radiation (Reference 51).

4.3.1.2 FEP Teflon

TEP was developed to provide for a genuine thermoplastic with TFE's properties. This material is the copolymer of TFE and hexafluoropropylene (HFP). HFP may be diagrammed as below.

The copolymer fluoroethylene-propylene (FEP), then, is diagrammed as:

Thousands of these copolymers are connected together in the solid FEP material. Pure FEP tends to be more nearly colorless and transparent than TFE. FEP made with the aid of wetting agents has a tendency, however, to look more brownish than TFE because it is less permeable, and therefore the wetting agents do not escape as readily during the sintering process as they do from TFE. Also, FEP is sintered at lower temperatures than TFE so there is less active volatilization of the wetting agents.

FEP, like TFE, is supplied by Du Pont both as a solid (in pellets) and in a dispersion. Unlike TFE, however, the FEP can be melt-processed since it is thermoplastic. Extrusion, injection molding, and vacuum forming are common methods of making parts of FEP. It can also be dip coated onto many different substrate materials and heat sealed, either to itself or to TFE.

The structural nature of FEP is more definite than that of TFE because the average crystallinity of FEP whi h has not been thermally degraded is essentially independent of variations in processing. In other words, crystallinity will be between 40 and 57 percent regardless of the cool down rate after sintering. The average molecular weight is unchanged from that in the resin unless excessive sintering temperature are reached.

When chilled from +68° to -100°F, FEP shrinks about 3.1 percent in volume, or about 28 percent less than TFE.

FEP film is noticeably more prone to pinholing, an occurrence of small fractures due to folding, than TFE and is also more subject to cold flow. TFE can be used at higher temperatures than can FEP, but FEP retains its flexibility better at low temperatures. FEP is also less permeable than TFE.

Work on propellant expulsion bladders has revealed that FEP is much more prone to solvent-stress-cracking than is TFE (Reference 60). N_2O_4 , MMH, Freon TF, heptane, and isopropyl alcohol were the fluids used in the tests. MMH had the least effect, closely followed by the alcohol. Some evidence may be interpreted to indicate that TFE's resistance decreases as the crystallinity increases.

FEP may be slightly less resistant to radiation damage than TFE.

4.3.1.3 Filled Teflon Materials (Reference 102)

Many fillers have been added to the basic Teflon resirs to provide a special purpose material, which exhibits an improved property, usually degrading some other property not as important for that application. Some of these improvements are:

- Increased wear resistance
- Increased creep resistance
- Increased thermal conductivity
- Reduced thermal expansion
- Change in electrical properties.

Fillers commonly used with Teflon include: glass fibers, carbon and graphite, metals (primarily bronze), metal oxides (primarily lead oxide and cadmium oxide), and others such as ceramic materials and molybdenum disulphide.

The choice of filler material is not simple since the performance of specific fillers cannot always be predicted. Empirical evaluation is generally required with the filler in the planned application to determine suitability. Table 4.3-4 provides some mechanical properties data on some filler materials.

It is always considered advantageous to minimize the filler content commensurate with other requirements. The following tabulation identifies some properties and indicates the limits of a potential filler material.

Property		Limit in Filler Material
Heat stability	-	Filler must withstand sintering temperature of Teflon TFE (approx. 750°F).
Particle shape		
Fibers and platelets	-	Maxir am reinforcement by overlapping but may result in anisotropic properties.
Irregular shapes	-	Irregular and angular provide maximum mechanical bond, but may be abrasive.
Spheres	-	Minimum chance of abrading but also minimum reinforcement and mechanical bond.
Particle size	-	Large particles result in a non-homogeneous mixture and possible variation in properties.
		Small particles may coat the PTFE preventing good adhesion between Teflon particles.
		Thermal and electrical properties also may be influenced by particle size.
Particle surface finish		This affects adhesion and bonding. If a good crosslinking agent is present, improved wear and creep properties would result.

Property

Limit in Filler Material

Particle structure and Physical properties

- The Tellon and filler mixture will have properties between the constituents.
 - To reduce creep a fiber with high modulus and high tensile strength required.
 - To retain low friction a ductile filler with low shear rate and low surface activity required.
 - To reduce wear not established but good thermal conductivity is a factor.

Chemical properties

- Determines chemical resistance to composite and could influence wear.

Other properties

It is undesirable for:

- The filler to agglomerate readily since it makes blending difficult.
- The filler to be hygroscopic since it leads to problems in mixture.
- The filler to react with Teflon TFE.

4.3.1.4 Teflon Data

The data provided in the following tables and figures is basic data on Taflon properties. Since Teflon is well characterized, references are available to obtain specific data and are provided in this section.

In Table 4.3-4 the basic mechanical property data is shown for Teflon TFE, FEP and two types of filled Teflon. This table provides a good comparison of the various Teflon types and the differences between them. Table 4.3-5 provides PV factors for Teflon, indicating capabilities and limitations for sliding or relative motion applications. Some references for additional and more comprehensive data are shown in Table 4.3-6. Using these references most Teflon characterization data may be obtained.

The figures provided, 4.3.1 through 4.3.12 provide additional basic data. In 4.3.1 the stress strain properties of Teflon TFE in air are shown, indicating the good strength properties over a wide comperature range.

Figure 4.3.2 provides the same information for Teflon FEP indicating generally a lower strength level for the same strain particularly in comparison at elevated temperatures. Figures 4.3.3 through 4.3.6 indicate the degree of deformation which Teflon TFE and FEP will experience under a tensile load at temperatures of -65, +75, +212 and $392^{\circ}F$ (FEP $347^{\circ}F$). These data are provided as a function of applied stress resulting in data useful for evaluating the effect of applied loads when subjected to temperature variations. In Figures 4.3.7 and 4.3.8 the total deformation under compressive load at +75 and +212 $^{\circ}F$ as a function of applied stress is provided. The degree of recovery or resilience after an applied compressive load is shown in Figures 4.3.9 and 4.3.10 at +75 and +212 $^{\circ}F$.

Table 4.3-7 provides an indication of propellant compatibility of Teflon with N_2O_4 and UDMH. Since much of the basic compatibility work with Teflon was accomplished some time ago, most existing data are in the form of summary properties rather than specific numerical data. This is primarily because Teflon was proven to be the most propellant compatible polymer generally available. Although, as shown in Table 4.3-7, there is some scatter in data and results, the conclusions generally indicate that little permanent change has resulted from propellant exposure.

Reference 199, summarizes other testing as follows in N_20_4 for 70 days at $75^{\circ}F$ Teflon exhibited:

- o 2 to 3% volume increase
- o 0.5% weight increase
- o 20% hardness loss
- o 11% increase in elongation
- o 11.5% reduction in tensile strength

After 20 days in $N_2^0_4$ Teflon FEP exhibited:

- o 1.6% volume increase
- o 0.4% weight increase
- o 38% hardness reduction
- o 2.9% increase in elongation
- o 20.5% reduction in tensile strength

For both Teflons, the changes in physical properties took place within the first two days of immersion, after that time all other changes were nearly zero (with the exception of the FEP, Teflon loss of hardness which was linear with time). In the same reference, it was noted that during an investigation of potential bladder materials, immersion of Teflon TFE and FEP in N $_2$ 0 $_4$ at 275 0 F for periods up to 88 hours resulted in approximately 7% strength reduction and up to 50% change in elongation. It was also noted that the N $_2$ 0 $_4$ washed out particles of Teflon which caused the N $_2$ 0 $_4$ to turn "milky".

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As indicated by these data, Teflon is a good polymeric structural material with a high degree of inertness. Its value has been shown by the number of applications shown in Tables 4.3-2, 4.3-3 and 4.3-4. There are limitations and deficiencies as indicated by mechanical and propellant data.

The major deficiency is the tendency to "cold flow" or continuously deform under load. Although some data provided in Reference 30 and presented in Figures 4.3.9 and 4.3.10 indicate some degree of recovery after an applied load is removed, most application information shows a high degree of permanent set, approaching 100% under high loads or elevated temperatures or a combination of conditions. Another problem area when used as a seal is a tendency to be contamination sensitive. The tendency is less than with a hard material, but is pronounced in a system exposed to contaminants. The capability to absorb contaminant particles is limited.

A major factor in the use of Teflon for sliding applications is the tendency for Teflon to flake. These flakes become contaminants and may become partially embedded in the seal, creating a leak path. The tendency for Teflon particles to be washed out as a result of N_2O_4 exposure also creates a potential contamination problem to the seal or other system components. Although there are many advantages to the use of Teflon, offsetting disadvantages indicate the desirability of replacing it with another material for long term, multi-use applications.

Table 4.3-4 Mechanical Properties of Teflon and Filled Teflon Resins (Reference 30)

n""	15% Graphite	1,300-2,700 185 203,000 2.6 55	1,080 4 7×10 ⁻⁵ in/in	6	0.08-0.10		
Filled "Teflon"	15% Glass Fiber	2,800-3,600 1, 325 312,000 2.7 54	1,000		0.10-0.13 0.	· · · · · · · · · · · · · · · · · · ·	
	FEP	3,000 300 95,000 2.9 D55		1.8 1.8	2.12-2.17		24,000 19,000 14,000 9,000 4,000 1,800 1,000
Unfilled Resins	TFE Fine Powder	2,500-3,500 300-600 40,000-90,000 D50-65		2.4	; ;		
	TFE Granular Resin	1,000-4,000 100-200 50,000-90,000			0.05-0.08	eratures (psi)	19,000 16,000 11,500 7,700 3,800 1,800 1,300
	Property	f (psi) F (psi)	ω +D	expansion per "F(/3"-140"F) Deformation under load 78°F, 1000 psi, 24 hr.(%) 78°F, 2000 psi, 24 hr.(%)	Static coefficient of friction against polished steel Specific Gravity	Yield Strengtn at Various Temper	-420° -320° -200° -100° - 68° 32° 73° 158°

Table 4.3-5.

Teflon and Selected Filled Teflon PV Factors
(Reference 30)

Parameter	FEP	TFE	15% Glass Fiber	15% Graphite
PV Limit (lb./in. ² x ft./min.)				
10 ft./min.	600	1,200	10,000	10,000
100 ft./min.	800	1,800	12,500	17,000
1,000 ft./min.	1,000	2,500	15,000	28,000
PV for 0.005 in. radial wear in 1,000 hr. (nonlubricated)	<10	20	3,100	1,500

Table 4.3-6 Sources of Properties Data for Teflon

Property	Reference	e Number*
	Teflon	FEP Teflon
Stress-Strain Diagram, Tensile	93, 94, 95	93, 94
Stress-Strain Diagram, Compressive	93	93
Stress-Strain Diagram, Shear	93	93
Modulus of Elasticity, Tensile	94, 99	94
Modulus of Elasticity, Compressive	94, 99	94
Shear Modulus or Modulus of Rigidity	94	94
Poisson's Ratio	93	93
Ultimate Strength, Tensile Ultimate Strength, Compressive Ultimate Strength, Shear	93, 94, 99, 101 94, 99	93, 94, 102 94
Yield Strength, Tensile	93, 94, 99, 101	93, 94
Impact Strength or Toughness	93, 94	93, 94, 102
Creep, Tensile	93	93, 102
Creep, Compressive	93, 95, 99	93, 102
Creep, Shear	93	93, 102
Relaxation, Tensile	93	93
Set, Compressive	99	30
Elongation at Rupture or Break	94, 99, 101	93, 94
Recovery, Tensile	93	93
Recovery, Compressive	93, 95	93
Faligue, S/N Curves or Endurance Limit	93	93
Abrasion Resistance	93	102
Wear Factors	93, 99	93
Friction Coefficient, Static	93, 99	102
Friction Coefficient, Dynamic	93, 99	93, 102
Hardness	93, 99	102
Permeability	97	97, 102
Density	93, 95, 99, 101	93, 102
Thermal Expansion/Contraction	93, 94, 95, 99, 100, 101	93, 94, 100, 102
Thermal Conductivity	93, 95, 99, 101	1, 10, 93, 102
Specific Heat	93, 95, 98, 101	1, 10, 93, 102

Table 4.3-7 Effects of Propellant Exposure on Teflon TFE (Ref. 210) $\rm N_2O_4$ Exposure (Temperature 70-75°F)

	Er josure	Time	Tensile	Elastic Modulus	Tensile Elastic Elongation Modulus	Volume	Weight %	Weight Hardness % Shore	Remarks
Teflon TFE	Controls		2550	55.8	0.3	001	100	290	
	Splash		2620		0.4	7.66	100.001	290	No visible change
	Fumes		2420		0.4	100.001 9.001	100.001	260	No visible change
	Liquid	1 Month	1640	31.2		103	102.6	490	No visible change
		3 Months 1630	1630	20		103.8 102.5	102.5	430	Very slight yellow
		6 Months 2300	2300		0.4	101.8 103.1	103.1	53D	Light yellow

UDMH Exposure (Temperature 70-75°F)

Teflon	Controls		2550	55.8	0.3	001	100	290	
	Splash		2080		9.0	100.4 100	100	590	No visible change
	Liquid	1 Month 1530	1530	41.9		99.1	99.1 99.99	260	No visible change
		3 Months				76.66			Very slight yellow
		6 Months 1440	1440	58.5		101.5	101.5 100.04	57D	Very slight yellow
		9 Months 1510	1510		0.5	1.001 6.001	100.1		Very slight yellow

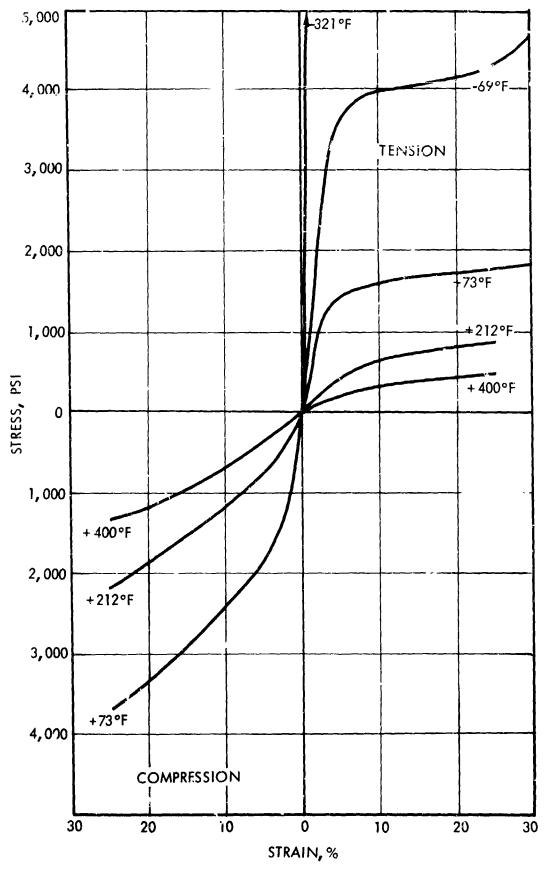


Figure 4.3.1 Teflon TFE Stress-Strain in Air (Ref. 30) 4-101

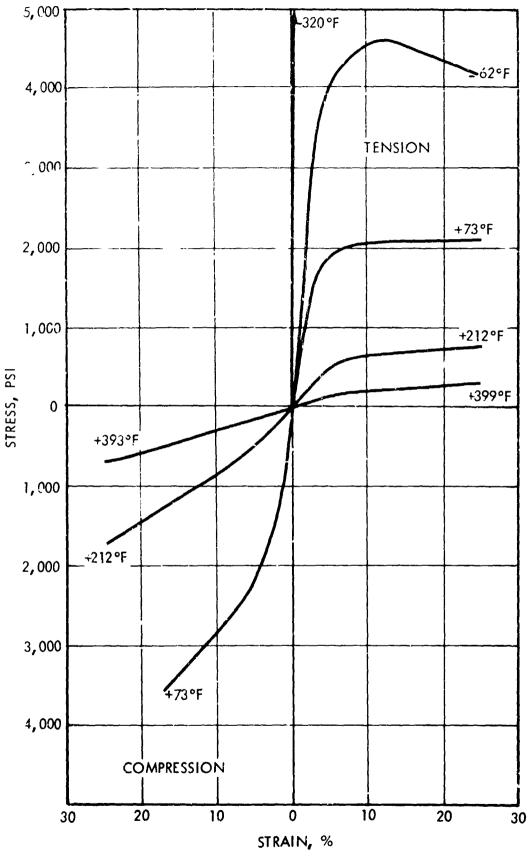
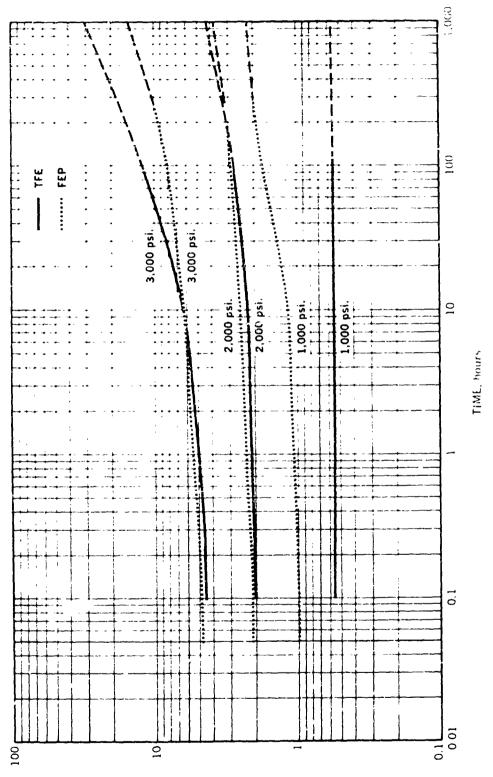
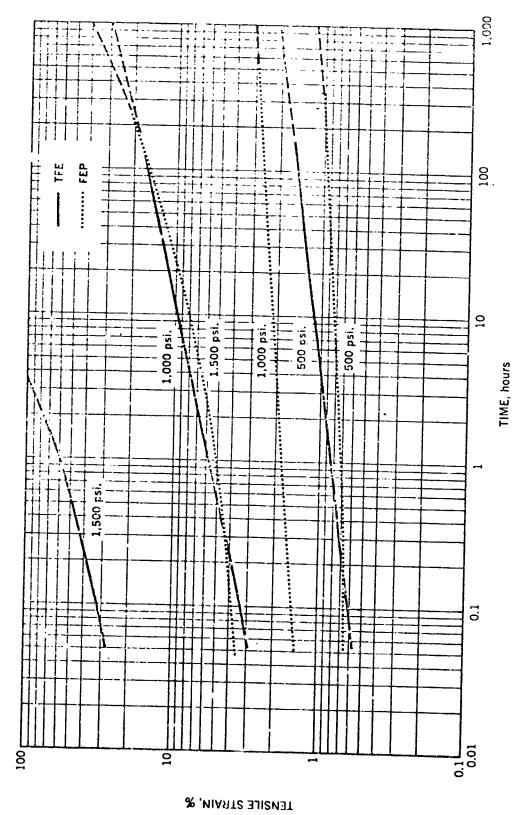


Figure 4.3.2, Teflon FEP Stress-Strain in Air (Ref. 30)

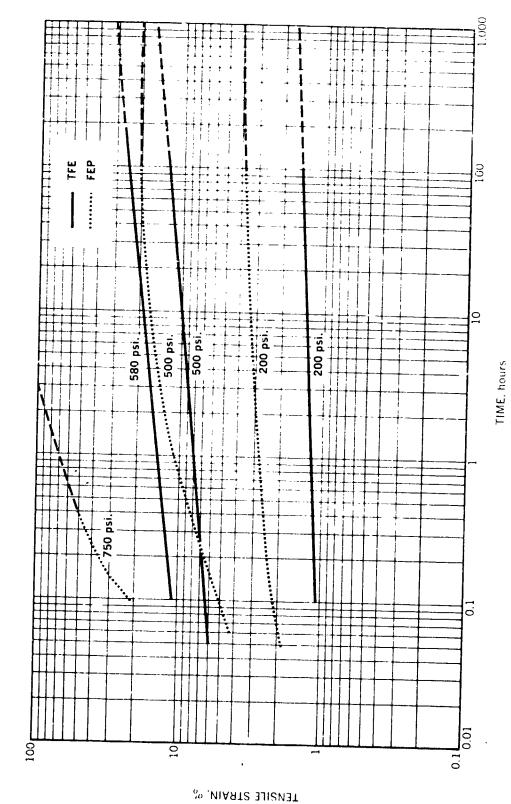


Teflon TFE Total Deformation versus Time Under Tensile Load at -65°F (Reference 30) Figure 4.3-3.

TENSILE STRAIN %

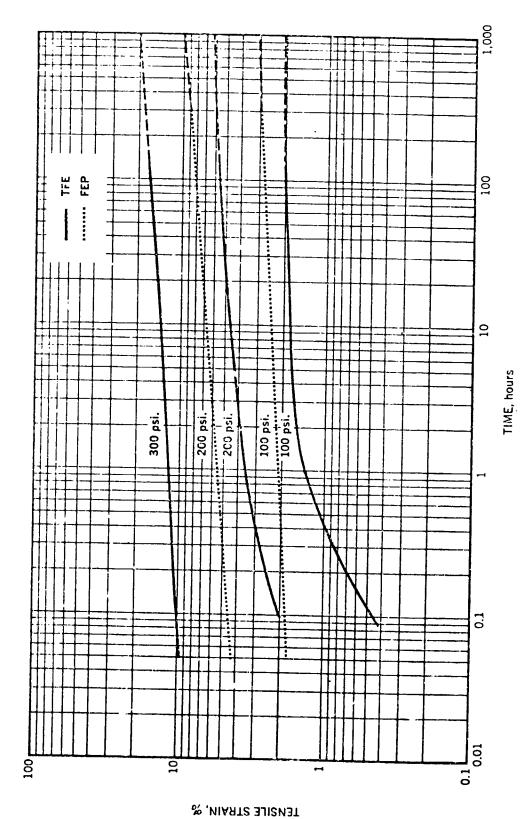


Teflon TFE and FEP Total Deformation versus Time Under Tensile Load at +73°F (Reference 30) Figure 4.3-4.

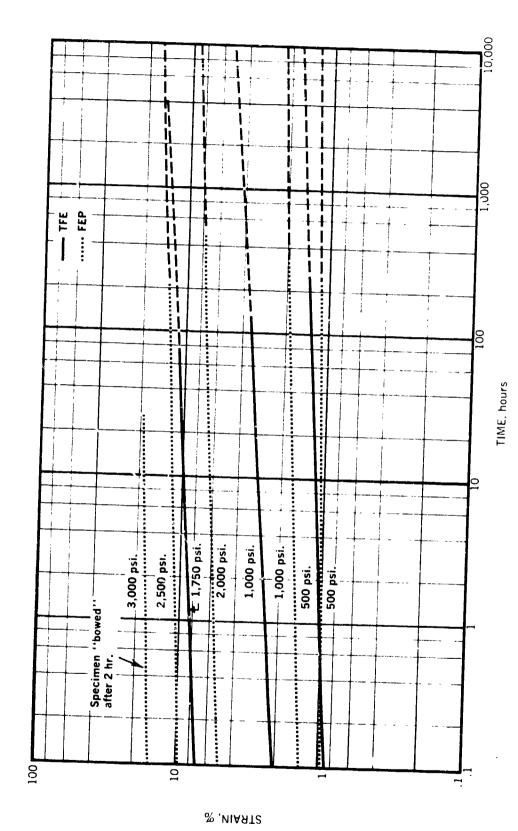


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Teflon TEE Total Deformation versus Time Under Tensile Load at 212°F (Reference 30) Figure 4.3-5.

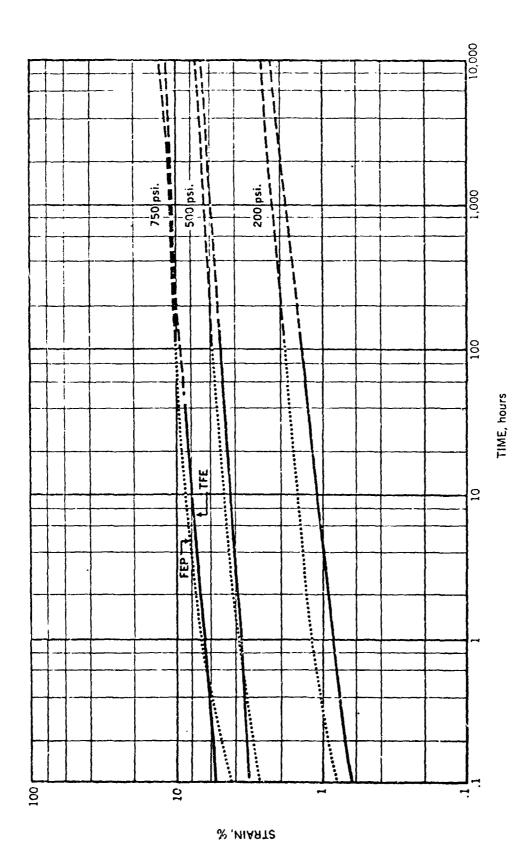


Teflon TFE and FEP Total Deformation versus Time Under Tensile Load at Elevated Temperature TFE 392°F. - FEP 347°F (Reference 30) Figure 4.3-6.



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Teflon TFE and FEP Total Deformation versus Time Under Compressive Load at 73^oF. (Reference 30) Figure 4.3-7.



Teflon TFE and FEP Total Deformation versus Time Under Compressive Load at 212^{0} F (Reference 30) Figure 4.3-8.

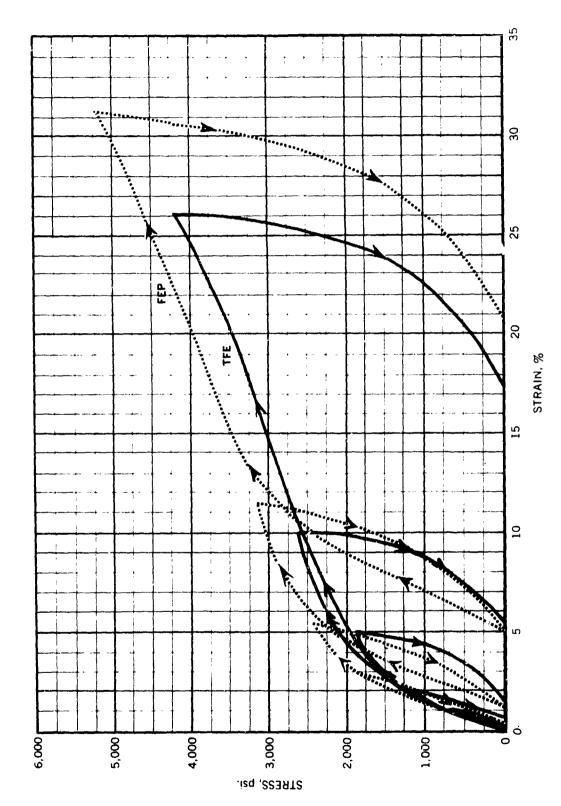


Figure 4.3-9. Teflon TFE and FEP Compressive Recovery at 73°F (Reference 30)

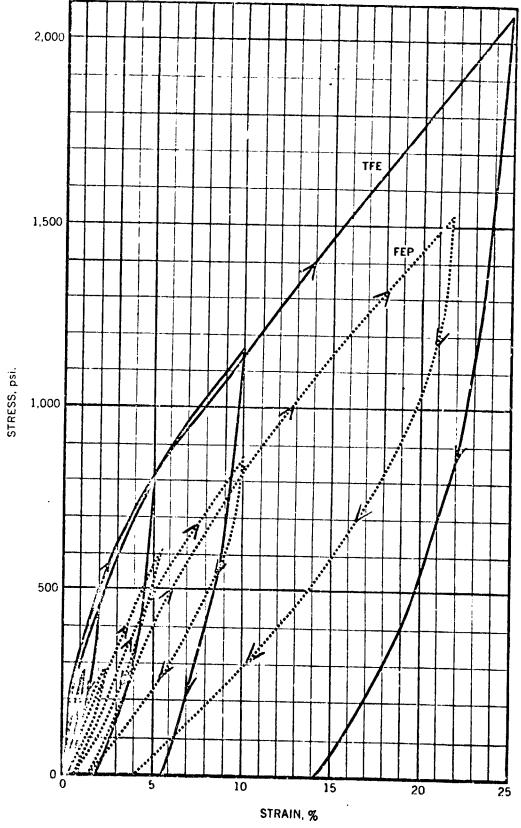
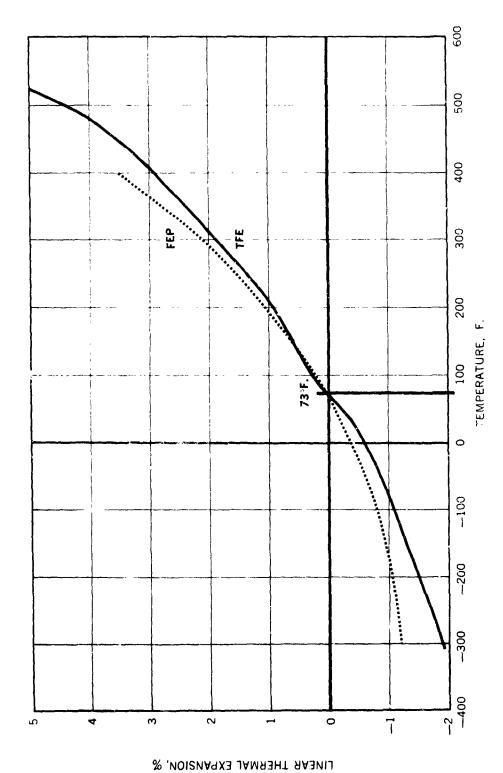
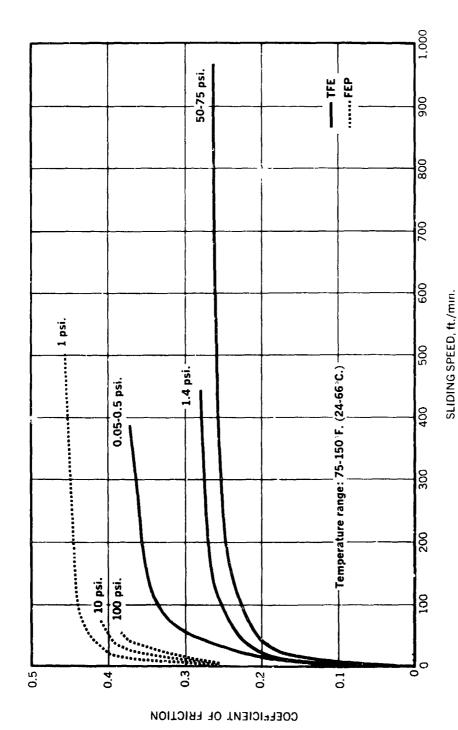


Figure 4.3-10. Teflon TFE and FEP Compressive Recovery at 212°F (Reference 30)
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Figure 4.3-11. Teflon TFE and FEP Linear Thermal Expansion versus Temperature (Reference 30)



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Figure 4.3-12. Teflon TFE and FEP Coefficient of Friction versus Sliding Speed (Reference 30)

4.3.2 Kel-F*

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Polychlorotrifluoroethylene (PCTFE) is commonly called Kel-F but this also is a registered trademark, in this case belonging to the Minnesota Mining and Manufacturing Company. PCTFE is marketed by several suppliers under their own trade names.

Kel-F is the same molecule as TFE except that every fourth fluorine atom on the backbone chain of carbon atoms is replaced with a chlorine atom. The Kel-F monomer (CF₂CFC1) may be diagrammed as below:

This inclusion of the chlorine atom is beneficial in its effects on mechanical, rheological and optical properties. However, it is somewhat detrimental in its effects on chemical, electrical and frictional properties and thermal stability.

The degree and kind of crystallinity in a given sample of Kel-F is a function of its thermal history. Quick-quenched resin tends to be more amorphous thar slow-cooled resin. Only relatively thin samples can be quenched rapidly and completely enough to inhibit crystal growth. In all cases some crystal nuclei develop even when molded or extruded parts are cooled rapidly. Prolonged aging at 300-390°F will increase the crystallinity of relatively amorphous samples.

At higher degrees of crystallinity, Kel-F is a denser, less transparent material with higher tensile modulus, lower elongation, and greater resistance to the penetration of liquids and vapors. The "amorphous" plastic, on the other hand, is less dense, more elastic, optically clear and tough.

^{*}Portions of this section are taken from Reference 100 and refer to Kel-F 81 plastic.

Molecular weight plays a vital role in the crystallization process. Long-chain molecules are slow to form crystal nuclei and reluctant to rearrange themselves into large spherulites. Consequently, components made from high molecular-weight Kel-F are strong and tough and therefore retain amorphic properties even after extended aging at high temperatures. In contrast, parts produced from resin degraded to low molecular weight during processing are susceptible to excessive crystallization and embrittlement. Molding conditions must be carefully controlled, therefore, to make sure that finished parts retain to the fullest extend possible all the properties inherent in the resin.

Kel-F is the rmally stable well above its melting point of 414°F. Compression molding at 500°F causes little or no degradation, whereas injection molding and extrusion at much higher temperatures (up to 650°F) degrade the resin to some extent. In this connection, it is important to distinguish between reversible changes which occur in the resin through heat treatment, i.e., through "quick quenching" or "slow-cooling," just below its melting point, and permanent changes which may occur because of overheating above the melting point (chain scission or degradation to a lower molecular weight).

Since it is a true thermoplastic, Kel-F can be fabricated by ordinary means, but high temperatures (500° to 600°F) and specially designed equipment are required. The relatively high molding and processing temperatures used to fabricate Kel-F components generate internal stresses. In many cases, stress relief of the plastic before finished machining and use is advisable to assure dimensional stability with age and temperature variations.

Kel-F has better strength than TFE below 210°F, and much greater resistance to cold flow than TFE. Its impact strength is good, some degrees of crystallinity giving as good or better values than some TFE samples.

Although Kel-F is swelled (but not attacked) by some chlorinated solvents at high temperatures, it is completely unaffected by most chemicals, including most concentrated acids. Stress-cracking has been a problem, however, under some conditions.

The threshold dose of radiation for a given degree of degradation in Kel-F is about five times that for FEP.

Table 4.3-8 provides basic mechanical properties of Kel-F 81 plastic. In Tables 4.3-9 and 4.3-10 the permeability and chemical resistance to fluids are indicated. No effect of UDMH exposure for 7 days at ambient temperature is seen while $\rm N_2O_4$, as expected, causes appreciable changes. Solvents do not adversely affect Kel-F with the exception of Trichloroethylene, which causes permanent dimensional and weight change.

Reference 199 states that Kel-F (300) exposed to $\rm N_2O_4$ at 75°F for 70 days became very soft, showed a 6% volume increase, 72% loss of strength and a hardness loss of 76%. Therefore, Kel-F is not recommended as a seal material, particularly for $\rm N_2O_4$ service. Although superior to Teflon in some areas, primarily cold flow, and strength up to 210°F, it retains other deficiencies including a low degree of contamination tolerance thereby, in general, resulting in a power seal material than Teflon.

Other Kel-F applications indicate (Ref. 71) that after 4 hour exposure to N_2O_4 , Kel-F strength is reduced to that of Teflon. In addition, exposure to amine fuels caused cracks and checks which damaged the sealing surfaces. In another case, Kel-F seals in amine fuel turned black and became brittle. In another application (Ref. 71), Kel-F used as a butterfly valve seal in N_2O_4 weakened after N_2O_4 exposure such that the operational life was essentially limited to one cycle.

Table 4.3-8 Kel-F (31) Plastic Mechanical Properties (Reference 100)

Property	Temperature °F	Crystalline	: Amorphous
Tensile Properties			
Tensile Strength (psi)	77 77 158	4630 5200 3550 540	4650 5260 2900 575
Elongation (%)	258 77 77 158	120 125 330	160 180 330
Yield Point (psi)	258 77 77 158	> 400 5200 5300 2700	> 400 4800 4700 1600
Yield Strength (psi) 0.2% Offset	258 77 77 158 258	560 2450 3350 1600 350	340 2000 2600 1100
Modulus of Elasticity Tensile (psi)	77 77 158 258	190 x 103 190 x 103 97 x 103 20 x 103	157 x 10 ³ 160 x 10 ³ 55 x 10 ³ 6 x 10 ³
Flexural Properties Flexural Strength (psi)	158	9600 5070	3600 3150 700
Modulus of Elasticity Flexure (psi)	258 77 158 258	1700 238 x 10 ³ 133 x 10 ³ 37 x 10 ³	185 x 103 79 x 103 15 x 103
Compressive Yield Strength - 0.2% Offset (psi)	77	5500	
Modulus of Elasticity Compression (psi)	77	170 x 10	
Shear Strength (psi)	77	5400	5600
Deformation Under Load 24 Hrs/1000 psi (%) (%)		0.20 0.40 4.00	0.40 7.12 > 25.00
Heat Deflection 66 psi 264 psi		258°F 167°F	

Table 4.3-9 (Ref. 100)

Permeability of Kel-F (81) Plastic

	$\left(\frac{\text{cc-cm}}{\text{sec - cm}^2 \text{ cmhg}}\right)$
+32 77 122	$.05 \times 10^{-10}$ $.30 \times 10^{-10}$ $.91 \times 10^{-10}$ 21.7×10^{-10}
	77

Table 4.3-10

Kel-F Propellant and Solvent Resistance (Ref. 100)

(7 Days at 77°F)

	Crystalline		Amorphous	
	% Weight Change	% Volume Change	% Weight Change	% Volume Change
Nitrogen letroxide (41°F)	8.2	9.4*	9.9	10.5*
UDMH	0	0.8	0.1	ე.1
Freon 113	0.9	0.6	0.9	0.4
Ethanol, 95%	О	-0.8	0	-0.7
Trichlorethylene	1.8	1.5	3.0	1.9
Water (21 Days/77°F)	0	0	0	0
*Material turns rubbery	1			

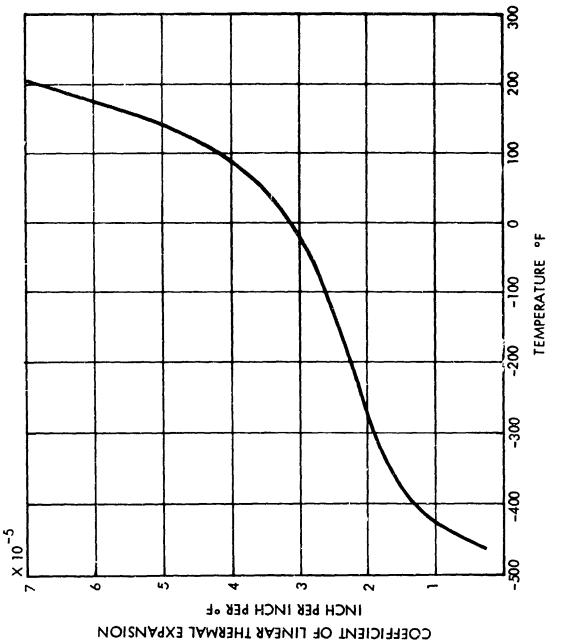
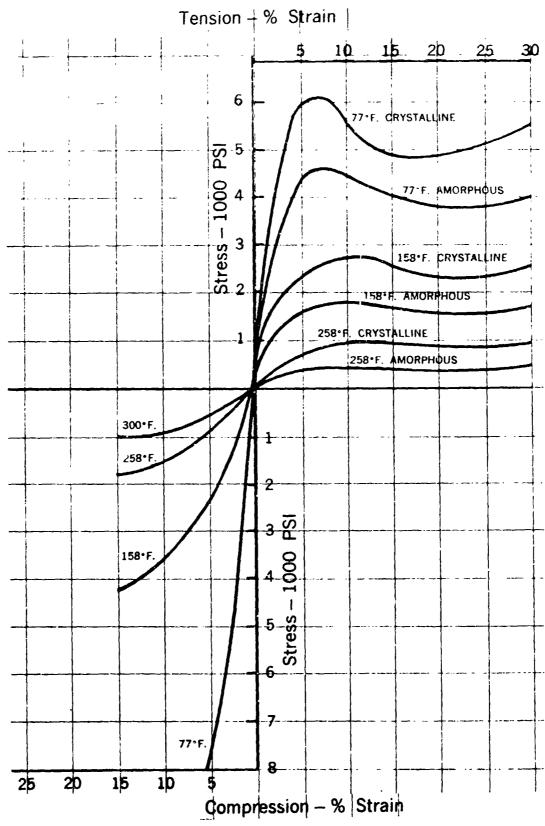


Figure 4.3.13 Coefficient of Linear Thermal Expansion for Kel-F Plastic (Reference 100)

4-118



Stress Strain Curves for Kel-F Plastic (Reference 100) Pigure 4.3.14

4-119

4.3.3 Kynar (Reference 103)

Kynar is a fluorinated thermoplastic resin, a high molecular weight polymer of vinylidene fluoride. Kynar contains 59% fluorine and has the chemical structure shown below. Kynar is a registered trademark of the Penwalt Corporation, Philadelphia, Pennsylvania.

This polymer is characterized by mechanical toughness, thermal stability, resistance to gamma radiation and resistance to many chemicals and solvent. It can be processed by compression molding, injection molding and extrusion among other techniques. Kynar is resistant to most acids and bases but the degree of affect is dependent on application, stress levels, etc. Strongly polar solvents such as Ketones and esters cause partial solvation, with increased effect at elevated temperatures.

The basic physical properties of Kynar (Grade 18) are shown in Table 4.3.11. These properties generally indicate a good structural plastic capable of fallication and load-application unsupported. As with many molded or extruded polymer resins, the properties of any given part is highly dependent on quality of forming and location in the bar as fabricated part since the edge effects can be appreciable.

A brief indication of propellant and cleaning solvents effects are shown in Table 4.3-12, for a 30 day 122°F exposure.

Other propellant tests (Ref. 167) indicate the following: In N_2O_4 at 150°F for 48 hours, Kynar film properties degraded to 73% of original tensile strength, and 10% of original elongation. In this case the color of the Kynar changed from tan to green. At room temperature for 13 months in N_2O_4 no change in tensile properties were noted, but the color changed to white. Hydrazine exposure for the same period resulted in some embrittlement and color change to yellow. The permeability of 7 mil Kynar film is reported as 95 to 100 mg/mil-day-atmosphere-in² for exposure periods of 48 and 100 hours. The rate for 21 hours is greater, (160 mg/mil-day-atmosphere-in²) being the initial saturation period.

Figures 4.3.15, 4.3.16, and 4.3.17 provide additional air exposure properties data. Cold flow resistance for crystalline resins at 2000 psi stress is very good as noted by 10,000 hours test data (Ref. 103). Increased load or amorphous resin reduces this resistance. Linear thermal expansion and TGA analysis data are provided in Figures 4.3.16 and 4.3.17. There is considerable variation in reported results for Kynar after exposure to propellants. Undocumented application data, particularly in test facility applications have shown Kynar deterioration through embrit-tlement and surface cracking after exposure to N_2O_4 for extended periods of time under stress. Valve seal chipping has been experienced under these conditions. At elevated temperature the effects would be expected to accelerate.

Reference 199 also indicates marked deterioration of Kynar when exposed to N_2O_4 at 275°F. Although some data indicate minimum effects, there are sufficient indications of severe attack to conclude that Kynar is not recommended for use in propellant systems and should be avoided in systems exposed to propellant vapors for extended periods of time.

4.3.4 Viton (Ref. 106)

Viton is a family of fluoroelastomers manufactured by E. I. du Pont de Nemours & Co. (Inc.). The name Viton is a trademark of that company. This elastomer family provides a range of combined heat and chemical resistance, but is not suitable for use with propellants.

Du Pont lists the continuous service limits as 3 years at 400°F; 3000 hours at 450°F, 1000 hours at 500°F, 240 hours at 550°F and 48 hours at 600°F. There are presently eight types of Viton. These are Types A; A-35; A-HV; B; B-50 and E-60C which are considered general purpose, d Viton LM and C-10 which are specialty types.

Viton A

Viton A is a general purpose high temperature and fluid resistant polymer with a Mooney viscosity in the range of the more conventional synthetic elastomers. Viton A processes well and is used in making molded and extruded 0-rings, gaskets and seals.

Table 4.3-11. Physical Properties of Kynar (Grade 18) (Reference 103)

PROPERTY	· VALUE
Tensile strength, 77°F (psi)	7000
Tensile strength, 212°F (psi)	5000
Elongation, 77° (%)	300
Elongation, 212°F (%)	400
Yield point, 77°F (psi)	5500
Yield point, 212°F (psi)	2500
Creep, 2000 psi, 77°F, 10,000 hrs. (in/in)	0.02
Compressive strength, 77°F (psi)	10,000
Modulus of elasticity, 77°F	
in tension (psi)	1.2x10 ⁵
in flexure (psi)	2.0x10 ⁵
in compression (psi)	1.2x10 ⁵
<pre>Izod impact, notched, 77°F (Ft-Lb/In)</pre>	3.8
<pre>Izod impact, unnotched, 77°F (Ft-Lb/In)</pre>	30
Durometer hardness (Shore D)	80
Heat distortion temperature, 66 psi (°F)	300
Heat distortion temperature, 264 psi (°F)	195
Abrasion resistance, Tabor CS-17, 1/2 kg load (mg/1000 cycles)	17.6
Coefficient of sliding friction to steel	0.14-0.17
Thermal coefficient of linear expansion	8.5x10 ⁵
Thermal degradation temperature (°F)	> 600
Melting point, crystalline (°F)	340
Specific gravity	1.76

Table 4.3-12. Effects Of Propellant A. Solvent Exposure on Kynar (Reference 103)

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Fluid	Tensile Strength After Exposure (PSI)	
Nitrogen Tetroxide	6500	
UDMH	4700	
Hydrazine	5800	
Trichlorethylene	6700	
Ethanol	6000	1

- Specimen is 5 mil film
- Exposure Period 30 Days at 122°F.
- Control Specimen 7200 PSI Tensile

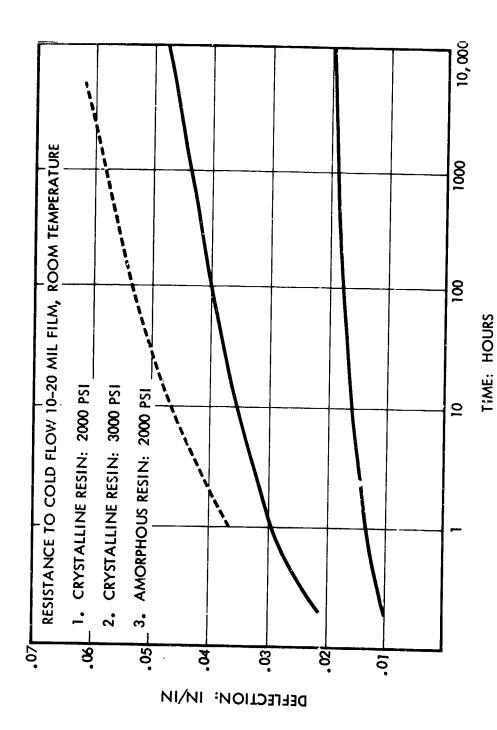


Figure 4.3.15 Kynar Cold Flow Resistance (Reference 103)

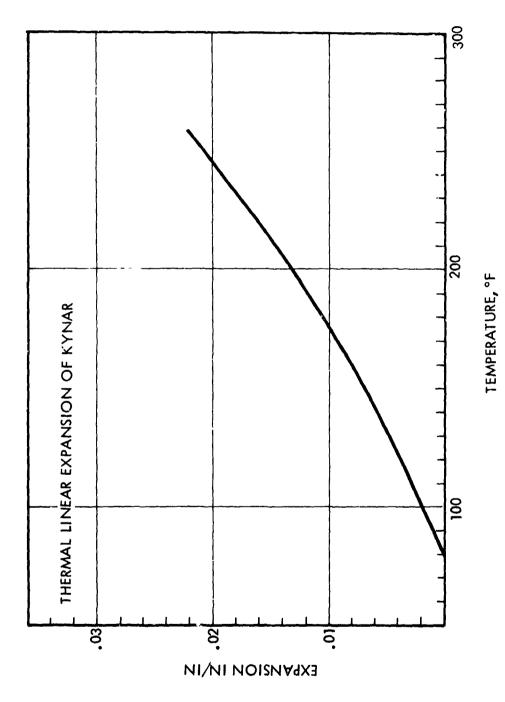


Figure 4.3.10 Kynar Linear Thermal Expansion (Reference 103)

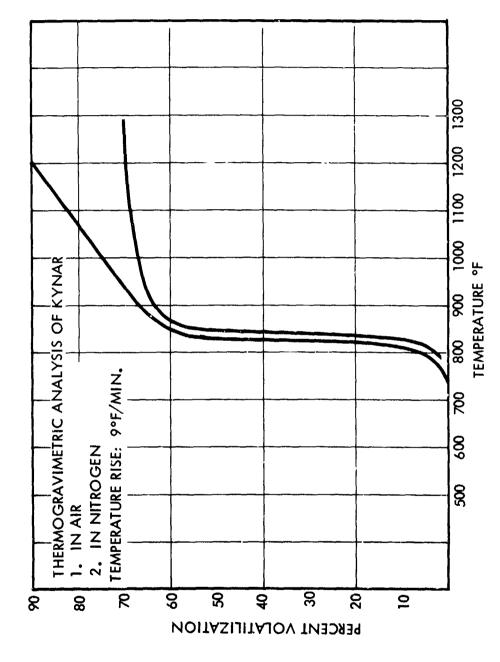


Figure 4, 3, 17 Kynar TGA Analysis (Reference 103)

Viton A-HV

Viton A-HV, is a high viscosity grade of Viton A. Due to its high molecular weight, it produces vulcanizates with improved hot strength and better resistance to compression set. Viton A-HV is particularly useful in high-pressure, high temperature sealing applications.

Viton A-35

Viton A-35 is a lower viscosity grade of Viton A and is, therefore, safer and easier to process than Viton A. In general, vulcanizates of Viton A-35 have tensile strength and modulus values 10 to 20% lower than Viton A, but other properties including heat resistance, and tear strength, are quite similar.

Viton B

Viton B is a Viton polymer with better heat and fluid resistance than the A-types, and a Mooney viscosity and processing characteristics intermediate between those of Viton A and Viton A-Hv. Both Viton A and Viton B are resistant to long term aging at 400°F but each reacts in a different manner to such exposure. Viton A is superior in retention of tensile strength but Viton B is better in retention of elongation.

Viton B-50

Viton B-50 is a low viscosity grade of Viton B. Compounds of Viton B-50 are safer processing, smoother extruding and have better mold flow characteristics than those of Viton B. The original tensile strength and elongation of some formulations based on Viton B-50 may be somewhat lower than those of Viton B but the heat and chemical resistance of Viton B-50 is equal to that of Viton B.

Viton E-60C

Viton E-60C is a new polymer which offers increased resistance to high temperature compression set. In addition, Viton E-60C has all the advantages of Viton E-60 the base polymer and superior compound storage stability, processing safety, and fast cure rate. Viton E-60C can be cured in thick sections.

VITON LM

Viton LM is one of two specialty types of Viton that have been developed to meet certain specific needs in the area of heat and fluid resistant polymers. Viton LM is a very low viscosity grade of Viton designed as a processing aid for the higher viscosity polymers. The use of small quantities of Viton LM in a compound of Viton A or Viton B improves the molding and extrusion properties of the compound with some sacrifice in vulcanizate performance. Heat resistance, chemical resistance and compression set resistance of vulcanizates containing Viton LM as a processing aid are affected to some extent.

Viton C-10

Viton C-10, the second of the specialty types, is a very low viscosity Viton that is particularly useful in caulks, sealants, high solids coatings and other applications where high concentrations of polymer in solution are required. Compositions based on Viton C-10 have the same fluid resistance and thermal stability after curing that are normally associated with the higher viscosity Viton polymers.

Most system sealing applications appear to use Viton A or B, with E-60C increasing in use as application data is increased. Viton E-60C is relatively new but, for systems experiencing high temperatures appears to be a good selection. Table 4.3-13 summarizes properties of the various Vitons and Figures 4.3.18 thru 4.3.22 provide additional data on Viton A, B and E-60C. Tables 4.3-14 and 4.3-15 provide additional Viton Data.

Viton materials are not normally used or recommended for propellant service although Table 4.3-14 indicates some tests with N_2O_4 . The results indicate a degree of incompatibility which would be unacceptable, since degradation occurs at room temperature in 7 days.

Viton is made from monomers including vinylidine fluoride (- CH_2 = CF_2 -) which has "readily oxidizable" hydrogen atoms compared with the fluorine atoms, thereby promoting degradation in oxidizers. Also, because of the

vinylidine fluoride which has F next to H atoms, the H is activated (i.e. becomes more acidic) becoming acidic enough to react with basic hydrazine, particularly as the temperature is increased.

The primary use and proper application is in hydraulic systems, and systems (non-propellant) exposed to elevated temperatures or other adverse conditions. Viton compounds should not be utilized in Space Shuttle storable propellant systems or systems exposed to propellant vapors for extended periods of time.

Table 4.3-13. General Properties of Various Viton Compounds (Reference 106)

Composition and Parameters		Comp	Compound and Properties	Propertie	Ş		
	A	A-35	A-HV	В	B-50	E-00C	
Viton	100	901	90	100	100	100	
MT Carbon Black	20	20	50	50	20	30	
Maglite Y	15	35	15	12	15	! '	
Maglite D	!	1	1	!	;	m 4	
Calcium nyaroxide DIAK #1	بر ! -	,	 ! !	t !	; ;	D !	
DIAK #3	: :	- 1	!	3.0	3.0	!	
DIAK #4	-	ł	2.0	!	;	!	
Physical Properties (Original)							
100% Ms. psi	1050	950	1000	650	220	006	
Tensil angth, psi	2450	1900	2600	2575	2300	1800	
Elongarton, %	160	160	200	410	250	200	
Hardness, Durometer A	75	77	_	72	89	77	
Physical Properties (af 70 hours at 528°F)		-					
100% Modulus, psi	1200	1300	1350	009	575	500	
Tensile Strength, psi	0061	1525	1900	2000	0061	1400	
Elongacion, & Hardness, Durometer A	28	2. 2. 2. 2. 2. 3. 3. 3. 3. 3. 3. 3. 3. 3. 3. 3. 3. 3.	85	35	700	6/2	
Compression Set, Method B, % (1 in. x 0.139 in	. 0-rings)						
70 hours at 75°F	21	23	18	19	23	ۍ د	
/U hours at 392°F 336 hours at 392°F	608	67 95		62 85	71 94	بر س دا	
						; 	

Table 4-3-14. Propellant and Solvent Effect On Viton A or B (Reference 110)

Flutd	Type of Viton	Tenstle Retained (%)	Elongation Retained (%)	Yolume Increase ⟨₡⟩	Hardness Change (Durometer)
Nitrogen Tetroxide (75°F) (7 days)	80	-58 -58	110	4.2	8
Freon il3 (75°F) (28 Days)	Ą	38	75	18.0	9
Alcohol, Ethyl (75°F) (7 Days)	¥	.26	100	1.7	+5
Trichlorethylene (75°F) (7 Dajs)	A	!	i i	19.0	;
Water, Distilled (212°F) (10 Days)	¥	86.5	104	6.0	7
Water (212°F) (3 Days)	₹	100	123	2.1	0

Table 4.3-15. Mechanical Properties of Viton E-60C (Reference 105)

			Сошро	Compound Number	_		
Physical Properties	Base Properties	B-3	C-3	D-3	E-3	F-3	6-3
Initial Tensile Strength (psi) 100% Modulus (psi) Elongation (%) Hardness (75°F) Duro. A	1725 875 190 79	1825 950 175 80	1650 850 180 79	2050 1000 170 78	2125 1200 160 78	1825 700 185 77	2125 925 180 77
After Elevaced Temperature Exposure	(70 hours a	t 528°F)					
Tensile Strength 100% Modulus Elongation Hardness	1375 475 270 80	1425 550 230 83	1300 300 280 80	1600 525 240 82	1625 800 230 82	1550 350 260 78	1675 350 260 80
Compression Set (%) Method B (0-Rings) 70 hours (14°F)* 70 hours (32°F)* 70 hours (39°F) 22 hours (392°F) 70 hours (392°F) 336 hours (392°F) 336 hours (392°F)	45 24 12 8 13 32	1 10 10 33	313112	1 10 128	33 33 1	1 1 6 1 2 8	

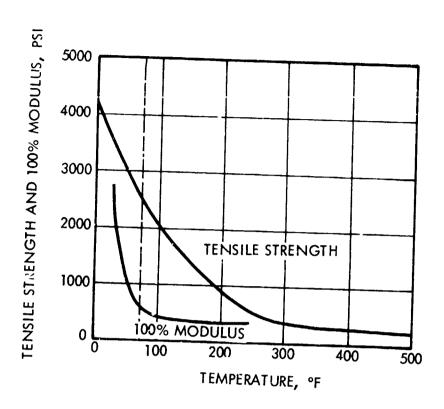


Figure 4.3-18. Viton A Mechanical Properties as a Function of Temperature (Reference 111)

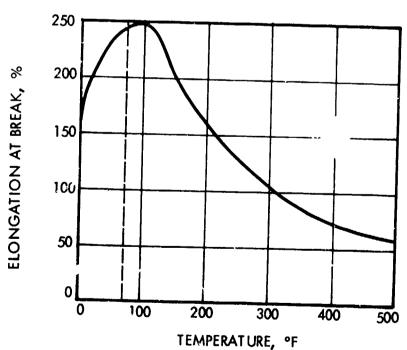


Figure 4.3-19. Viton A Elongation as a Function of Temperature (Reference 111)

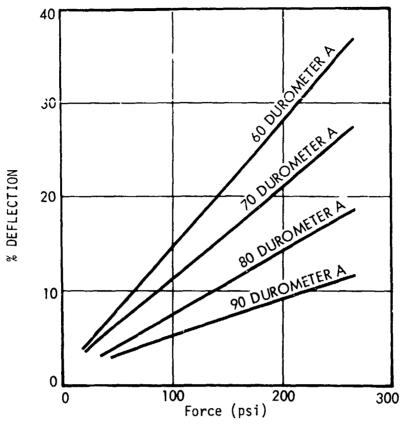


Figure 4.3-20. Viton B Compression Deflection Characteristics (Reference 111)

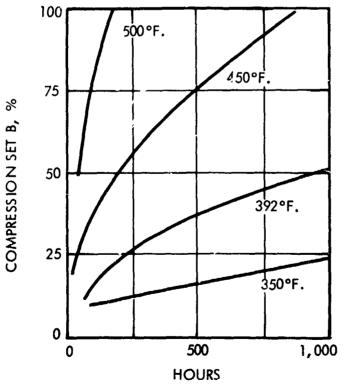


Figure 4.3.21 Yiton E-GOC Elevated Temperature Compression Set in Air (Ref. 105) 4-134

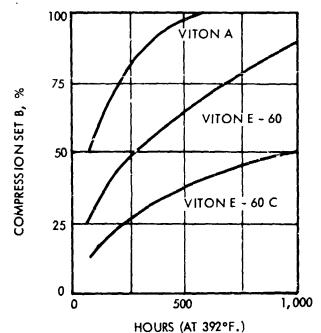


Figure 4.3.22 Viton A and Viton E-60C Long Term Compression Set (0-Rings) (Ref. 105)

4.3.5 <u>Vespel</u>

Vespel is a polyimide resin manufactured by E. I. du Pont de Nemours & Co. (Inc.) and is a trademark of that company. The material is characterized by toughness and heat resistance. The types of Vespel compounds are SP-1, SP-21, SP-22, SP-31, SP-5 and SP-211. Table 4.3-16 indicates the types and compositions of these various compounds. The manufacturer indicates an operating range to 900° F in some applications and a continuous service temperature of 500° F to 600° F in air. Cryogenic applications at -420F have indicated suitability for this temperature range. The filled Vespels are LOX impact sensitive, while SP-1 is nominally not LOX impact sensitive although some data are conflicting.

Table 4.3-17 summarizes the mechanical properties of each grade. The good friction and wear characteristics are shown in Table 4.3-18.

Table 4.3-19 indicates the effects of exposure to N_2O_4 and UDMH. As shown in this table both fuels and oxidizer attack Vespel and result in property degradations. Little data are available related to vapor applications but it can be concluded that some attack will occur.

The optimum application for this material would appear to be in inert gas service or with cryogonic media.

Figures 4.3.23 through 4.3.33 illustrate tensile strength, modulus of elasticity as a function of temperatures, as well as stress strain characteristics, deformation tendency, fatigue resistance and thermal characteristics. The most common grades are SP-1, SP-21 and SP-22 as shown in these figures.

Vespel is a good structural seal material but is not propellant compatible as shown in Table 4.3-19, and should not be considered for propellant systems. Potential applications could include gaseous systems and cryogenic systems. Some investigation has been conducted related to Vespel sealing properties at low temperatures. At ambient and higher temperatures this plastic exhibits lack of tolerance to contaminants typical of a plastic.

Table 4.3-16. Compositions and Characteristics of Vespel (Reference 112)

·		
Resin Designation	Description	Property Characteristics
SP-1	Unfilled base resin	Structural parts where maximum strength is required. High-temperature resistance.
SP-21	15 weight % (nominal) graphite	Graphite formulation for non- lubricated seals and bearings.
SP-22	40 weight % (nominal) graphite	Similar to SP-21 but offers 1/3 less thermal expansion.
SP-31	15 weight % (nominal) molybdenum disulfide	Non-lubricated seals and bear- ings in vacuum or dry environ- ments.
SP-5	42 weight % (nominal) short glass fibers	Precision mechanical parts requiring low thermal expansion.
SP-211	SP-21 base with 10% Teflon	Uniformly low coefficient of friction over a wide range of operating conditions.

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Table 4.3-17 Vespel Mechanical Properties (Ref. 112)

		1-dS	- i	SP-21	ا ئ	SP-22	22	SP-31	<u>.</u>	SP-5	
PROPERTY	Ė.	₫	Ŧ	. ₹	¥	. ₹	Ī	ž	: =	#	· =
MECHANICAL Tensile Strength (ultimate) (psi \times 10^3)	2	7.5	0.1	= = = = = = = = = = = = = = = = = = = =	9	7.6	5.5	2	4	;	,
	572	6.3	8 .	0 9	4.4		2.5	7.2		:	•
Tensile Elongation (ultimate) (%)	73 572	3.0	5.0	3.0	2.0	2.8	9.6	4.6	9.6 9.6	6.0	9.9
Tensile Modulus (psi κ 10^3)	572	3,485	450	95.55 55.05	365 365	790	310	545 315	305	1360	995
Flexural Strength (ultimate) (psf $_{\rm H}$ $_{\rm 10^3}$)	73	18.5 10.5	8.4	. 18 o	11.4	15.4	8.6	22.0	16.0	12.0	6.0
Flexural Strain (ultimate) (%)	73 572	12.7		7.1	3.5	3.0	2.1	8.6	5.7	1.2	9
Flaxural Modulus (ps. x 10^3)	73 572	460	\$ 50	360	308	889	385	534	495	1000	620
Compressive Strength (ultimate) (ps: \times 10 3)	73	36.7	45.0	29.0	¥.6.	18.2	18.6				
Compressive Strain (ultimate) (%)	73	×50, *05,	88	33.0	38.0 13.0	17.1 11.3	18.2				
Compressive Modulus (pst \times 10^3)	73	569	515 235	98 98	54C 290	380	909				
Axial Fatigua Endurance Limit (psi \times 10^3) at 10^3 cycles	73	<u>.</u>	 	8.3	5.4						
at 10' cycles	8 28	84.	3.8	3.8	3.4						
Flexural Fatigue Endurance Limit (psi \times 10^3) at 10^3 cycles at 10^7 cycles	22	5.6	:		-						
Shear Strength (psf \times 10^3)	73	12.3	13.7	6.01	11.6					•	
Impact Strength, Izod, notched (ft.lb./in.)	52	-	0.7	9.0	0.5						
Poisson's Ratio	73	0.41		6.4		-					
Confederation of Course Courses					_						
Controlent of Linear Expansion (microins/in./ ⁰ F)	73-572 80 to 73	3.58 2.48	7 ,9	. 23	% %	- 9	98	\$2		۔ ۔	. 12
Deformation under 2000 psi load (1)	122	0.14		0.10	-						
Deflection Temp , 264 psi (OF)	·	-680				-					
Mardness (Rocknell E scale)	73	45-58	32-44	32-44	-	15-40		40-60		44-56	-
Specific Gravity	73	1.43	_	1, 51		35 [5			·

Table 4.3-18 Coefficient of Friction and Wear Characteristics of Vespel (Reference 112)

Parameter	SP-1	SP-21	SP-22	SP-31
Wear Rate (unlubricated) (in./1000 hrs)				
in nitrogen (PV = 25,000)	0.010-0.015	0.004		0.05
in air (PV = 25,000)	0.25-1.2	0.09	0.06	0.26
Coefficient of Friction (steady state, unlubricated)				
in nitrogen (PV = 25,000)	0.04-0.09	0.06-0.08		0.05-0.08
in air (PV = 10,000)	0.49	0.35		
in air $(PV = 25,000)$	0.29	0.24	0.30	0.24
in air (PV = 50,000	0.23	0.10		
in air (PV = 90,000)	0.21		}	
in air $(PV = 100,000)$		0.07	0.09	
in air $(PV = 315,000)$		0.04	}	
Static in air	0.35	0.30	0.27	

Table 4.3-19 Propellant and Solvent Compatibility of Vespel (Reference 112)

		sile tention	Elonga % Rete		% Wt
Fluid	SP-1	SP-21	SP-1	SP-21	Change
Nitrogen Tetroxide (120 hrs at R.T.)	60	60	60	60	+3
HMGU	45	45	65	65	-

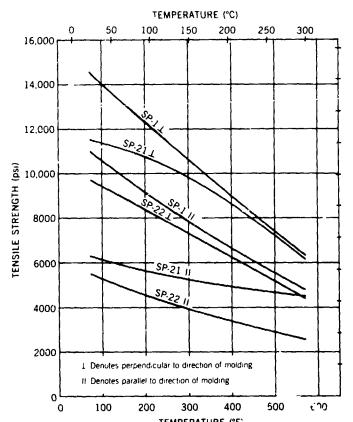


Figure 4.3.23 Vespel Tensile Strength as a Function of Temperature (Ref. 112)

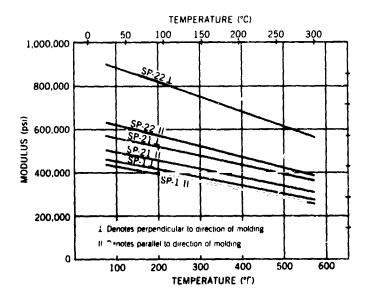
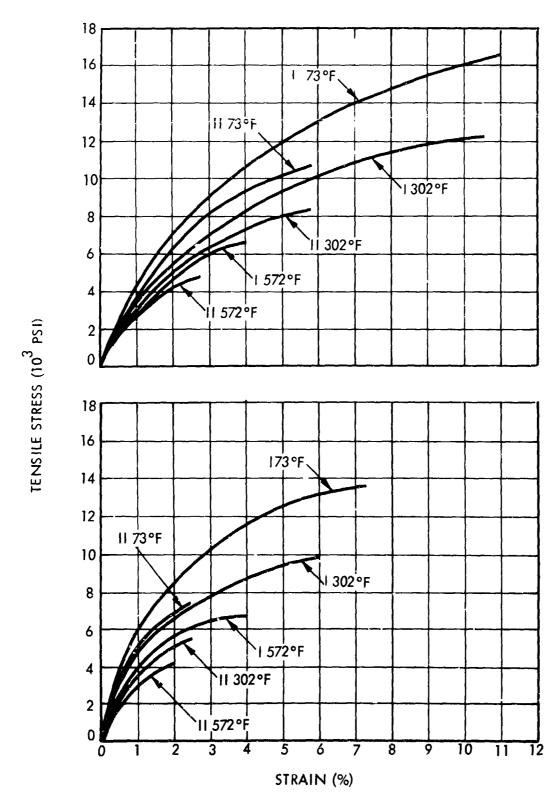
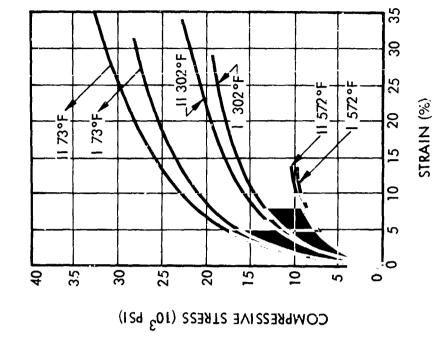


Figure 4.3.24 Vespel Modulus of Elasticity as a Function of Temperature (Ref. 112)



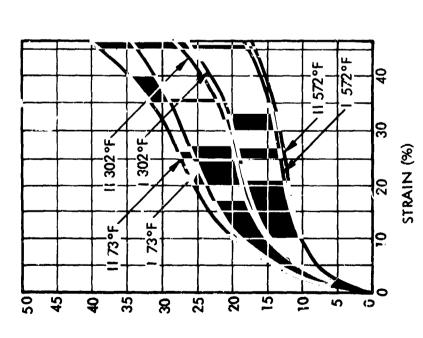
I DENOTES PERPENDICULAR TO DIRECTION OF MOLDING II DENOTES PARALLEL TO DIRECTION OF MOLDING

Figure 4.3-25. Vespel SP-1, SP-21 Stress Strain Curves in Tension (Reference 112)



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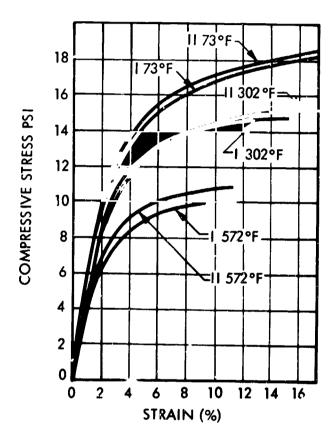


COMPRESSIVE STRESS (10^3 PSI)

DENOTES PARALLEL TO DIRECTION OF MOLDING

I DENOTES PERPENDICULAR TO DIRECTION OF MOLDING

Figure 4.3-26. Vespel SP-1 and SP-21 Stress Strain Curves in Compression (Reference 112)



- I DENOTES PERPENDICULAR TO DIRECTION OF MOLDING
- II DENOTES PARALLEL TO DIRECTION OF MOLDING

Figure 4.3-27. Vespel SP-22 Strees Strain Curves in Compression (Reference 112)

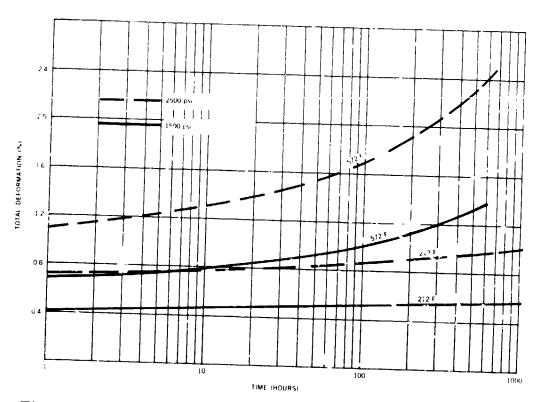


Figure 4.3-28. Vespel SP-1 Deformation as a Function of Time (Reference 112)

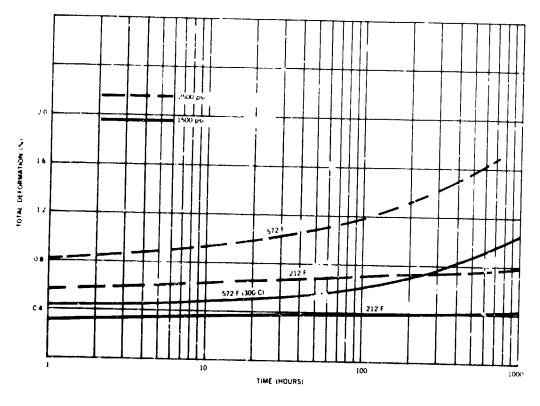


Figure 4.3-29. Vespel SP-21 Deformation as a Function of Time (Peference 112)

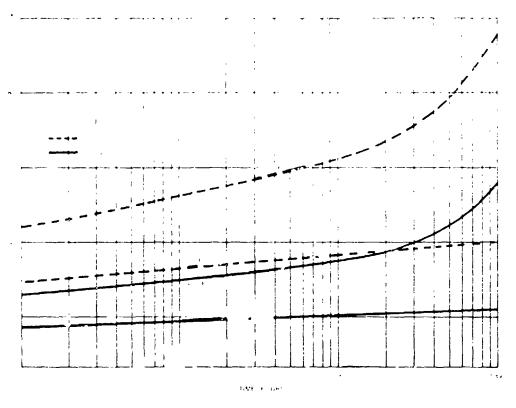


Figure 4.3-30. Vespel SP-22 Deformation as a Function c. Time (Reference 112)

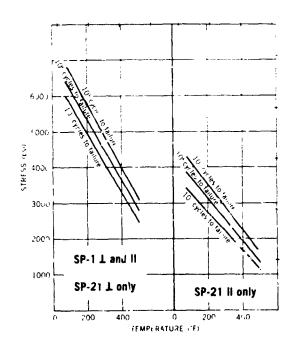


Figure 4.3-31. Vespel SP-1 and SP-21 Fatigue Resistance (Reference 112)

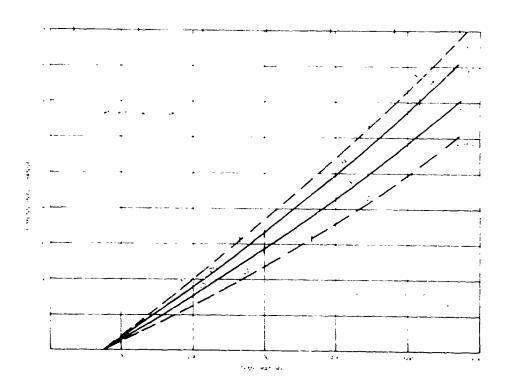


Figure 4.3-32. Thermal Expansion of Vespel SP-1 and SP-21 (Reference 112)

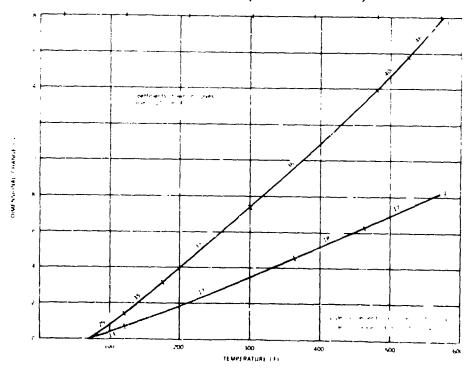


Figure 4.3-33. Thermal Expansion of Vespel SP-22 (Reference 112)

4.3.6 Carboxynitroso Rubber (CNR)*

CNR belongs to a family of elastomers known as nitroso rubbers. This family is represented by the structure shown in the following generic formulation:

$$\left[N-O-\left(\stackrel{l}{C}\right)_{\mathbf{x}}\right]_n$$

The first member of this family of polymers is the copolymer generally referred to as nitroso rubber. The synthesis reaction for preparation of this copolymer is shown below.

The outstanding characteristics of nitroso copolymer are its

solvent resistance, oxidizer resistance, low T_g (-60°F) and non-flammability (even in pure oxygen). The terpolymer is essentially the same as the copolymer with very small amounts of pendant perfluorobutyric acid. Terpolymers with various acid percentages have been studied and the most promising concentrations for use as an elastomer have ranged from 0.5 to 1.5 mole percent. The structure is shown below:

^{*}Most of this information is obtained from References 66 and 67.

Two types of cures are used, CTA (Chromium Trifluoroacetate) and DPD (Dicyclopentadiene Dioxide).

The suggested mechanism for CTA vulcanization is formation of the chromium salt of the polymer.

$$\frac{CTA}{(CF_2)_3}$$

$$C=0$$

$$C=0$$

$$C=0$$

$$C=0$$

$$C=0$$

The suggested DPD cure mechanism depicted shows only the first step in this process.

Although data from Peference 66 and others indicate a low compression set for both CTA and DPD cures, later application data indicate very high values, comparable to Teflon under the same conditions.

CNR is extremely non-flammable even in pure oxygen environments. Experiments comparing CNR and Teflon (by Thiokol) indicated that at pressures up to 600 psia in pure oxygen Teflon sheet samples could be ignited, while CNR sheet samples would not ignite or char.

The N2O4 and nitric acid resistance of properly cured CNR are among its more interesting properties. CTA cures appear to be the most resistant; however, DPD vulcanizates are resistant for at least 3 mouths and show much lower stress relaxation than CTA vulcanizates. Other epoxide cures show relatively poor resistance, degrading significantly within a few lays. Metal oxide vulcanizates are also generally inferior for this purpose. The only materials known to have a seriously degrading effect on the nitrosc terpolymer are amines. A prime example is hydrazine, which degrades the polymer in a matter of hours at elevated temperatures. Liquid fluorocarbons are frequently solvents or CNR gum and can be expected to cause a high degree of swell in the vulcanizate. However, some fluorocarbons cause little change; for example, Freen MF, frequently used as a flushing fluid in rocket propellant systems, causes only 19 percent volume swell in CNR vulcanizates. The composition and some mechanical properties or characteristics of CNR (AF-E-110. Resistazine 88) are shown below: (Ref. 66)

Ingredients	AF-E-110
CNR	100
Silstone 110	20
CTA	5
Mechanical Properties	
Tensile Strength (psi)	1600
Elongation (%)	150
Hardness, Shore A	85
Maximum Service Temperature in Air, Continuous Service	375°F
Bin Life (uncured)	>45 days

Mechanical Properties (Continued)	AF-E-110
Shelf Life (cured)	> 90 days
N ₂ O ₄ Volume Swell	
at 70°F	15%
at 165°F	45%
Safety Characteristics	Excellent
Qualitative Resistance to:	
H ₂ O ₂	Excellent
IRFNA	Excellent
Hydrazine	Poor
Freon MF	Excellent
Freon 113	Excellent
Hexane	Excellent
Toluene	Excellent
Petroleum Hydraulic Fluid	Excellent

A comparison of the permeability of CNR (AF-E-110) with Butyl and Teflon FEP is shown in Figure 4.3.34.

Work is presently in process by TRW Systems (Contract F33615-71-C-1233) oriented toward improvement of CNR by compounding and curing techniques. This is a continuing program and only interim data are available at this time. Various cure techniques have been investigated, the most successful being an Amide cure and an Ester cure.

The following is a comparison of mechanical properties obtained with the NT-5 (CTA) cure and the TRW cure.

MECHANICAL PROPERTIES OF CNR (1.0 Mole-%)

Property	<u>NT-5</u>	TRW
M _{10G} , psi	500	450
T _b , psi	975	800
E _b , %	325	300
Shore A Hardness	80	

Mechanical Properties of CNR (cont.)

Property	<u>NT-5</u>	TRW
Compression Set, %	100	85
(22 hours/160°F)		
Helium permeability, scc/cm ² -hr.	6.0	0.20
$(\Delta P = 15; 0.075-inch thick)$		
N ₂ O ₄ Permeability, mg/cm ² -hr.	0.23	0.055
$(\Delta P = 15; 0.075-inch thick)$		

It is obvious from these data that no major improvements have been made to date, except in the reduction of the permeability to helium and N_20_4 . The remaining properties are essentially identical.

From these data, it can be concluded that CNR is not a recommended seal material for Space Shuttle applications. In addition to the poor mechanical properties and in particular essentially 100% compression set after a relatively short period, availability would be a problem. No source of this material is known with the exception of the quantities TRW is making for the program mentioned previously.

In summary, there are no known advantages and a number of disadvantages in the use of CNR m_{α} terial.

4.3.7 Ethylene Propylene Terpolymer (EPT)

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Stillman Rubber Company Compound SR 724-90 is an EPT compound characterized by high resistance to degradation by hydrazine, and low compression set characteristics are characteristic of the better existing EPT compounds. The properties shown below are representative of this material. It is being successfully used in a number of flight monopropellant systems.

Another ethylene propylene compound currently in use is a proprietary compound of the Parker Sea! Company designated as £515-8. Parker Seal data (Ref. 207) states that it is suitable over a temperature range of -65 to +300°F and for vacuum service to 10⁻⁷ torr. The material hardness is Shore A 60. This material has been successfully used in hyarazine

Ethylene propylene terpolymer compounds have been used successfully in hydrazine base fuel systems, as previously indicated. There is a degree of degradation which occurs in physical properties that is progressive but at a lower rate with time, after initial exposure. Compression set also occurs with an increase with time, resulting in sealing problems of pressure drop variation.

It can be concluded that EPT compounds are suitable for fuel service, but are not optimum, and if used for extended service such as Space Shuttle, would be expected to cause problems in some cases.

An extremely important factor in the use of EPT compounds is control of material purity and exact compounding. Small amounts of impurities will seriously degrade compatibility as will compounds using carbon black as filler compounds. (Ref. 204)

Ethylene Propylene Terpolymer Properties

Parameter	SR 724-90 (Reference 92)
Tensile Strength, 22 ⁰ C, psi	1790
Elongation at Break, 22°C, psi	300
Shore A Hardness, 22°C	90
Tensile Strength After 100 Hours in 72°C Hydrazine, 22°C, psi	1300
Elongation at Break, 100 Hours in 72°C Hydrazine, 22°C, %	180
Tensile Strength, 100 Hours in 72°C Alcohol , 22°C, psi	1975
Elongation at Break, 100 Hours in 72°C Alcohol , 22°C, psi	220
Stress at 5% deflection, O.l in./min First Deflection, psi	325
Hysteresis Loss From 25 Deflections at 5%, %	15.5
Compression Set, 70 Hours at 72°C, %	22
Properties in Hydrazine	
Initial Deflection, Hanging Weight Test, %	7
100-Hour Creep, Hanging Weight Test, %	1.5
Permanent Deformation, Hanging Weight Test, %	3.3
Volume Swell, 24 Hours, 22 ^o C, %	3.0
Compression Set, 50-hr/72°C + 115-hr/22°C, Immediate Value, %	26.8
Compression Set, 50-hr/72 ^o C + 115-hr/22 ^o C, 24 Hour Rest, %	19.2

4.3.8 Butyl Rubber

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Butyl Rubber has been used extensively in hydrazine base fuel service. There are various compounds, each generally proprietary to the manufacturer. No data are presented here since it is not a present candidate for fuel service due to the availability of superior materials such as EPR and EPT.

An example of Butyl compound is Stillman Rubber SR 634-70, a proprietary Butyl compound of the Stillman Rubber Division, Sargent Industries. Stillman states that it is suitable for service over a wide temperature range and also for vacuum applications. Reference 63 reports a tensile strength of 2058 ps; and an elongation of 400 percent for a sample with a Shore A hardness of 471.

In general Butyl compounds tend to cause fuel decomposition and experience mechanical property degradation including high compression set over a period of time. Reference 199 indicates a high MMH decomposition at 275°F in 24 hours with 20% volume increase and a 10-12 Shore A hardness reduction. With available alternate elastomers there are no apparent advantages in using Butyl for propellant systems seals.

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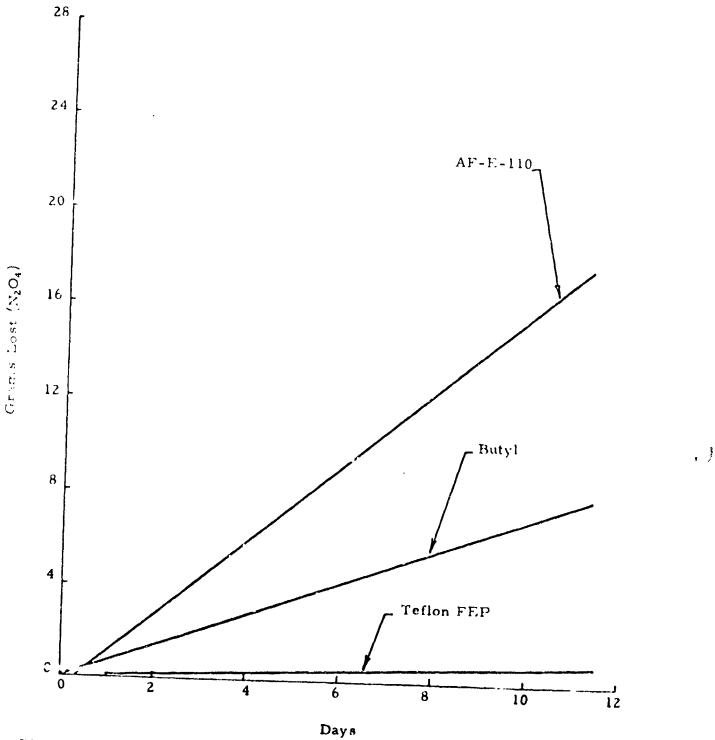


Figure 4.3-34. Permeability of CNR, Butyl and Teflon FEP to N_2O_4 at 65°F

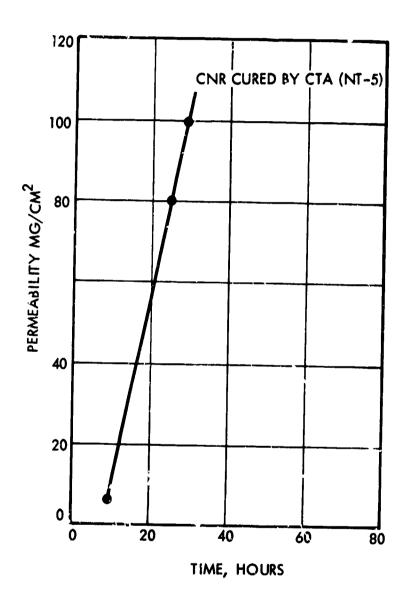


Figure 4.3-35. Helium Permeability of CNR (Reference 66)

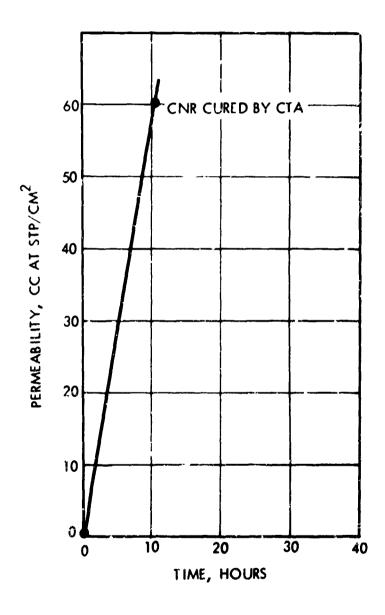


Figure 4.3-36. Dinitrogen Tetroxide Permeability of CNR (Reference 66)

5. Material Comparison

5.0 MATERIAL COMPARISON

The preceding sections have indicated some properties of available seal materials, and in most cases an assessment of their applicability for Space Shuttle. Properties and attributes of these materials vary widely, and will further vary with specific design applications. This section provides a comparison and assessment of these materials categorized as materials for oxidizer service and materials for fuel service. It providing these comparison data, point by point comparison is generally not possible since various manufacturers conduct different tests and report results differently. However, an overall comparison can be made to determine relative material suitability.

5.1 Seal Materials for Oxidizer Service

The only existing seal materials which are applicable to oxidizer service are AF-E-124D and Teflon. It is considered that of these two materials, AF-E-124D provides the greatest advantages for Space Shuttle applications. The advantages and disadvantages of these materials will be discussed later after a brief discussion explaining the exclusion of other materials which have been used in the past, particularly carboxynitroso rubber (CNR).

Other seal materials which have been used include CNR, Kel-F. Kynar and Butyl. The reason for using these materials in the past (with the exception of CNR) has been that no other material could come close to providing the required properties at that time.

CNR, although developed specifically for N_2O_4 service, exhibits very poor mechanical properties, the most significant being almost 100% compression set, which results in no inherent sealing capability. Other factors such as low tensile strength and a high permeability rate are not as important but are negative features. As a structural material for seals in N_2O_4 , this material cannot be recommended.

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Kel-F is a good structural material, but is only marginal in $\rm N_2O_4$ resistance and for long periods and/or elevated temperature in $\rm N_2O_4$ is poor. As indicated in the previous section, in $\rm N_2O_4$ for seven days at room ambient caused the material to change from a hard plastic to a rubbery consistency. After seven days under the same approximate conditions Kel-F 300 in addition to softening experienced a 72% loss in strength.

Butyl rubber should not be even considered for N_2O_4 service since it is completely incompatible. The only past applications have used good design techniques to extremely limit the area of exposure to N_2O_4 thereby reducing the rate of deterioration (Ref. 21). This approach is not necessary or desirable with present state-of-the art materials.

Although there appear to be some conflicting data related to Kynar N_2O_4 service, it can be concluded that Kynar is unsuitable for liquid N_2O_4 service. At elevated temperatures (+275°F) Kynar is severely attacked. The degree of attack is lower at room ambient conditions but attack occurs over longer periods of time causing embrittlement, surface cracking and discoloration. No vapor exposure data are available, but extended exposure to N_2O_4 vapor would be expected to degrade Kynar properties at a lower rate and without specific supporting test data, this application would also not be recommended.

5.1.1 Teflon and AF-E-124D

Some of the properties of these two materials are compared in Table 5-1. Since Teflon properties are, to some extent, dependent on processing, crystallinity, and void content, a range of values exists. These values are not design values but typical of the resin properties and will also vary with configuration. However, in general, the ambient temperature tensile strength of AF-E-124D is in the same range as Teflon, with Teflon capable of 4000 psi, but both TFE and FEP are typically in the 2000 to 3000 psi range.

The elevated temperature properties differ somewhat in that the tensile strength of AF-E-124D is approximately the same as the yield strength of Teflon at +250°F to 300°F. Thus, in a tensile application neither should be used at these temperatures, with AF-E-124D experiencing tensile failure and Teflon yielding and elongating. In compressive applications, both can be used, but a high degree of compression set can occur, close to 100% in both cases.

The tensile strength of Teflon is significantly higher than AF-E-124D at elevated temperatures to approximately 500° F, and decreasing rapidly thereafter. Figure 5.1 shows these approximate relationships. It is interesting to note that the Teflon yield strength is always below the tensile strength of the AF-E-124D. Since the Teflon yield strength may be considered the design point for minimum cold flow it can be seen that the usable tensile properties are roughly equivalent except that continuous deformation occurs with Teflon and to a much less degree with AF-E-124D.

The static and dynamic coefficients of friction are similar, with AF-E-124D only slightly higher, an unusual characteristic for an elastomer. The basic advantage of AF-E-124D over Teflon is in the area of compression set and recovery after release from compression. For example, at equivalent strains of 25%, Teflon experiences a 17% set while AF-E-124D has no measurable set when compressed to 25% and released. The same is true for equal stresses. As an example, for a stress of 2500 psi, a strain of 10% is experienced with Teflon resulting in permanent set of 5%, while the same stress in AF-E-124D results in 45% strain and essentially zero permanent set. While these examples actually illustrate only the properties of elastomers vs. plastics, they do show a basic difference of these two materials.

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For long term loading, Teflon will approach a 100% set if unconstrained while AF-E-124D will recover to varying degrees depending on initial compression and time.

Table 5-1 Comparison of Seal Material Properties for $\rm N_2O_4$ Service

		MATERIALS	LS		
Property	AF-E-124D	Teflon TFE	CNR (TRW)	KYNAR	KEL-F
Tensile Strength (70-75°F) (PSI)	1900 to 2500	1000 to 4000	800	7000	4650
Elongation at Break (%)	200 +	100 to 200	300	300	160
Hardness	85-90 Shore A	50-65 Durometer	No Data	D 80	No Data
Elevated Temperature Tensile Strength (PSI)	250 (300°F)	1500 (400°F)	No Data	5000 (212°F)	575 (258°F)
Yield Strength (PSI)	N/A	200 (400 F) 500 (250°F)	N/A	5500 (77°F) 2500 (212°F)	2450 (77°F) 350 (258°F)
Coefficient of Friction-Static, Against Steel: (70-75°F)	0.08 to 0.11	0.05	No Data	0.14 to 0.17	No Data
Specific Gravity	2.05	2.1 to 2.3	No Data	1.76	No Data
Compression Set $(N_2O_4 200^{\circ}F)$ 22 hrs) %	52.5 (Method B)	86.3 (Method B)	85 (Method B) 160°F/22 hrs	N/A	N/A
Coefficient of Linear Thermal Expansion	45.8 × 10 ⁻⁵	5.5 x 10 ⁻⁵	No Data	8.5 x 10 ⁻⁵	4.2 × 10 ⁻⁵
Strength After Exposure to N204 - % Retained	R.T. 3 mos 89% 100 hrs/160°F 76%	R.T. 75 Days 88.5% 275°F/88 hrs. 93%	No Data	R.T 13 mus ~ zero charge 150°F/48 hrs 73% 275°F/88 hrs - "severe attack"	Material turns Rubbery (7 days at 77°F)
Availability	Limited Quantity	poog	Poor	Poog	Poc 9
Considered Suitable for N ₂ 04 use?	Yes	Yes	O.	NO	NO

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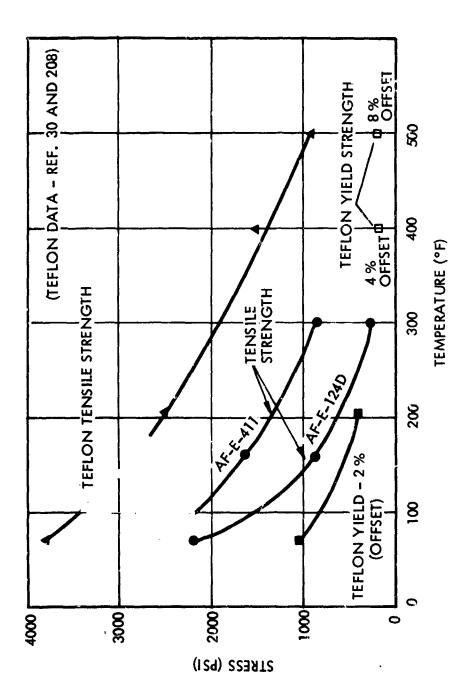


Figure 5.1 Comparison of AF-E-124D and Teflon Tensile Strength as a Function of Temperature

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Propellant compatibility and effects of propellant exposure are a function of temperature and time of exposure. In general, Teflon TFE and FEP are considered compatible with $N_2^{\,0}_4$ for service at ambient temperatures and up to 160°F. Reference 199 states the following: "After 70 days of immersion (at 75° F, dry $N_{2}O_{4}$), a 2 to 3% volume increase, a weight gain of 0.5%, a hardness loss of 20%, an 11% increase in elongation and a loss in strength of 11.5% occurred with TFE Teflon. After 20 days, the FEP Teflon showed a volume increase of 1.6%. a weight gain of 0.4%, a 38% loss in hardness, an increase in elongation of 2.9%, a loss in strength of 20.5%. For both Teflons, the changes in physical properties took place within the first 2 days of immersion, after that time all changes were nearly zero (the hardness loss of the FEP Teflon was an exception which was linear with time)." Both TFE and FEP Teflon showed losses in strength of about 7% (after immersion in dry N_2O_4 at a temperature of $275^{O}F$ for periods up to 88 hours) and changes in elongation up to 50%. It was also noted that N_2O_4 washed out particles of Teflon which caused the ${\rm N_2O_4}$ to turn milky. This reference further states that all rubbers (Butyl, EPR and Nitroso) either blistered. ignited or completely dissolved under those conditions and Teflon did fare better than the other non-metals.

All this emphasizes that $N_2^0_4$ service is extremely severe and any polymer subjected to this service is in a very severe environment.

Teflon data from this program has been primarily related to compression set characteristics after exposure to propellants. After N_2O_4 exposure at $200^{\circ}F$ for 22 hours, Teflon 0-rings experienced a set of 9.7% for an initial compression of 12% or 86.3% of the initial compression.

AF-E-124D under the same exposure conditions experienced a 14.7% set for an initial compression of 30% or 52.5% of the initial compression.

The long term compression set characteristics cannot be predicted statistically with the limited number of data points available. However an approximation of the long term trend may be obtained by plotting the available data and extrapolating the elevated temperature data to a time

period based on room temperature exposure. This is shown in Figure 5.2. The Arrhenius relationship was used here to get an approximate time based on elevated temperature, accelerated tests. While it is recognized that this method is not universally accepted and only useful over a limited temperature range and is at best a rough approximation, it is used here only as a tool, not as an exact relationship. The data points for 1 month at 160°F and 22 hours at 200°F were reduced to time at 75°F by using the approximate relationship that equivalent time is decreased onehalf with each 10°C (18°F) increase in temperature. The data in Figure 5.3 indicate a relatively flat slope between the data points. extrapolated value for 1 month at 160°F appears to indicate that under some conditions this degree of compression set could occur in ${\rm N_2O_4}$ over a long period of time. The time extrapolation accuracy as well as single point data must be taken into account in assessing this point. However, it can be concluded that some increase in compression set will occur as a function of time, but at a low rate. Normally, the maximum set would be considered to have occurred prior to the 9 month data point. Considerably more data are required to determine the rate and maximum amount of compression set.

Properties of AF-E-124D after 6 month exposure to N_2O_4 at ambient room temperatures (approx. 70°F to 75°F) showed a loss in tensile strength of 18%, loss of elongation of 3% and no change in hardness. These losses occurred gradually over the 6 month period, the values at 3 months being: Tensile loss - 11%, elongation loss - 0, hardness loss - 6%. This is comparable to the previous TFE values after 60 days of 11.5%; +11%; -20%. (Reference 199)

These problems are significant and while acceptable in past systems and operating durations, can present problems of sufficient magnitude to compromise some long term oxidizer system operations. While AF-E-124D has no operational experience, the basic attributes appear capable of resolving most of these problem areas.

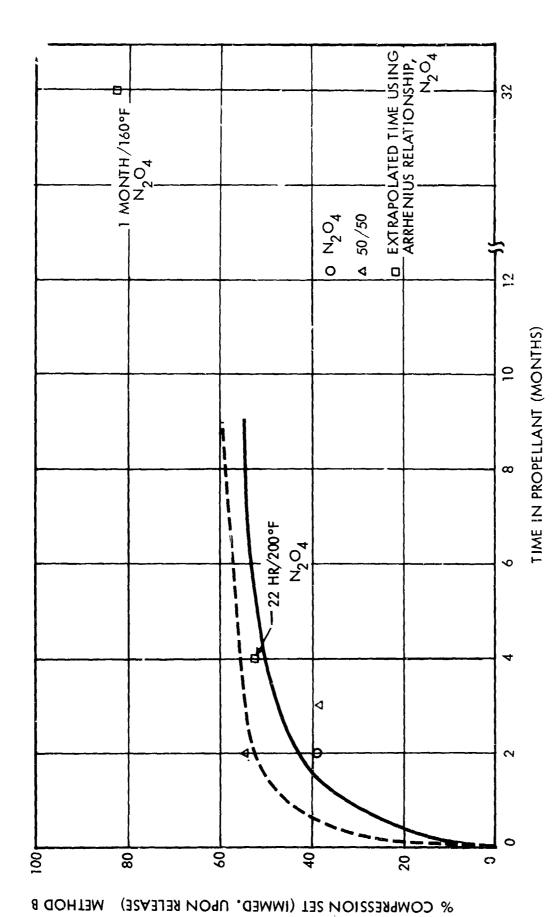


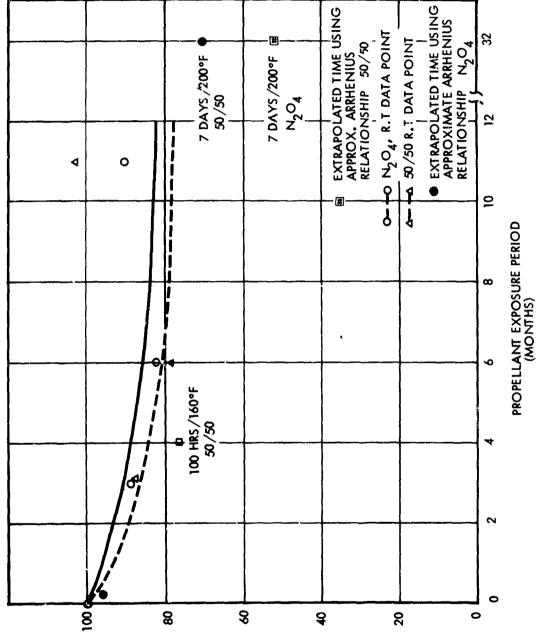
Figure 5.2 AF-E-124D Compression Set With Time

In summary, the major problems which have been encountered in the use of Teflon seals are:

- o Cold flow resulting in little sealing capability particulary in static seals.
- Flaking during relative motion applications such as ball valve closures, resulting in contaminants and leakage.
- o Particles of N_2O_4 have been washed out of Teflon at elevated temperature, 2a potential source of contamination.
- o In partially contaminated systems, particles of contaminant are embedded partially in Teflon, causing a leakage path.

The long term strength characteristics of AF-E-124D in N_2O_4 , also cannot be statistically evaluated with the limited number of data points available. However, a graphic representation of the data as shown in Figure 5.3 provides some indication. The 3 and 6 month data indicate a tensile strength reduction as expected. The 9 month point indicates a high degree of data scatter. No rationale exists for an increase in strength after N_2O_4 exposure and this point must be considered inaccurate due to test technique or other factors. However, since evaluation of the test method and data indicate no obvious error it must be assumed that this point is only slightly in error. Thus the tensile strength at 6 months and 9 months are considered to be approximately equal. It would be expected that some degradation in tensile strength with time would occur but at a low rate. The extrapolated data point for 7 days at 200°F (using the Arrhenious relationship) indicates a more rapid rate but since the time relationship can be questioned, it is probable that this reflects the maximum degradation over a very long period. As with compression set, much more long term effort is required to acquire data, and permit more accurate long term extrapolation by such means as the WLF relationship.

% TENSILE STRENGTH RETAINED AFTER PROPELLANT EXPOSURE



AF-E-124D Tensile Strength Retention With Propellant Exposure Time Figure 5.3

5.2 <u>Seal Materials for Fuel Service</u>

For fuel service a number of candidate materials are available. These materials include AF-E-411, AF-E-124D, Teflon, Ke1-F, Kynar, Ethylene Propylene Rubber and Butyl Rubber.

Although other materials are discussed in Section 4.3, they are not candidate materials for propellant system applications. Both Viton and Vespel are acceptable materials for pneumatic or hydraulic systems. Viton is particularly well suited for elevated temperature and very low compression set applications. Vespel is characterized by its toughness and heat resistance. Both cryogenic applications and elevated temperature applications are suitable for Vespel.

There are no conditions or applications where Vespel or Viton should be considered for propellant system service. Table 5-2 provides a comparison of some candidate seal material properties. Butyl Rubber is not included since properties and compatibility vary widely with formulation. However, Butyl exhibits common undesirable characteristics of hydrazine base fuel decomposition and degradation of properties with exposure to propellant. These result in a structurally weak material with high compression set, both undesirable in a seal material.

As shown in Table 5-2, the basic mechanical properties of each material are satisfactory with no marked differences other than those related to material type, plastic vs. elastomer.

The primary area of difference is the degree of compatibility with hydrazine base fuels. The summary assessment as discussed below is that AF-E-411, AF-E-124D and Teflon are virtually unaffected by fuel propellant exposure. EPR, Kynar, Butyl and Kel-F are affected to a much larger degree with a wide variation ir compatibility, with EPR being one of the more compatible in this group and Butyl and Kel-F being the less compatible.

Table 5-2 Comparison of Properties of Material; for Fuel Service

			MATERIALS			
		AE_E_1240	Teflon	Kynar	EPR	Kel-F
Property	AP-E-411	7- IV			0021	4650
(150) (1) 4+	2510	1900 to 2500	1000 to 4000	000/	2	
Tensile Strength (1 _b) (131) 70-75°F		250 (+300°E)	1500 (400°F)	5000 (212°F)	No Data	515 (258°F)
Elevated Temperature Tensile Strength (PSI)	1100 (+300°F)	- 000 - 000 - 000 - 000		5500 (77°F)	N/A	2450 (77°F) 350 (258°F)
Yield Strength (PSI)	N/A	N/A 85-90 Shore A		Shore D 80	Shore A 90	No Data
Hardness (70-75°F)	Calcilo De-68		Durometer		000	160
Flondation at Break (%)	170	+ 500	100 to 200	300	300	3
70-75°F		0 08 to 0.11	0.05	0.14 to 0.17	No Data	No Data
Coefficient of Friction-Static Steel, Air (70-75°F)	0.20 to 0.28		06	65%	76%	~ 100% (75°F 7 days UDMH)
T _b After fuel b Exposure (% Retained)	(3 mos/75°F,50/50)	5°F,50/50)	(1 mo/75°F,UDMH)	$(122^{2}F/30 \text{ days}, (102^{2}F/30 \text{ mg/4})$	(102 H/ N2H4)	
Compression Set Air (%)	9,5% 168 hrs/75°F	. 36% 100 hrs/77°F	:No Data - High	N/A	22%(162°F/ 70 hrs)	N/A
(Method b) Compression Set Fuel (%)	.39% 2 mos/75 F 50/50	54% 2 mos/75°F 50/50	No Data - High	A/A	26.8% [162°F/ 50 hr + 72°F/ 115 hr N ₂ H ₄]	и/А
Availability Service	Good	Limited-Batch Lot Yes	Good Yes	Good	Good	0N No
Sultable 101 lact 55: 115						;
Recommended for Optimum Fiel Service	Yes	No	ON	ON	ON	NO

In summary, suitable materials for use in hydrazine base propellants are AF-E-411, AF-E-124D and Teflon TFE in that order. EPR, properly componded, is usable but not recommended because of poorer overall performance. Materials which are rot considered for use and therefore not recommended are Kynar, Butyl and Kel-F.

Butyl rubbers tend to decompose hyd.azine propellant, causing increased system pressures where significant areas of Butyl are in contact with the propellant as in expulsion bladder applications. The Butyl rubbers have performed acceptably in some hydrazine systems even with this long term tendency to break down. Reference 199 indicates a 3 rating (doubtful compatibility) for Butyl rubbers in general.

Ethylene propylene rubbers are, in general, greatly improved over most Butyl rubbers. Although they do evidence some pressure rise in a closed container in hydrazine, the degree of disassociation of hydrazine is reduced, and mechanical property retention is better.

The mechanical properties of EPR (particularly Stillman Rubber Compound SR 724-90) compare favorably with AF-E-411 initially. Although the EPR properties are slightly lower.

The difference in materials is demonstrated after exposure to hydrazine. After 100 hour exposure at room temperature to hydrazine the tensile strength of EPR is reduced 24% and elongation at break is reduced 27%. AF-E-411 under the same conditions exhibits an increase in tensile strength of 11% with a 10% reduction in elongation.

A good measure of material compatibility is the hydrazine decomposition rate mentioned earlier: where Butyl exhibits 0.1 ps1 per day. EPR exhibits .0038 psi per day, (Ref. 199). The compatibility of EPR depends significantly on the compounding variations used, with carbon black compounds having a significantly higher rate of fuel decomposition than others.

Kynar is affected to varying degrees dependent on exposure. Because of a tendency toward degradation over the long term, when exposed to hydrazine base fuels, little application has been attempted due to the

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availability of other materials such as Teflon. As a hard plastic it exhibits similar seal properties to a soft metal and can be replaced by one. For areas requiring soft scals, EPR has been a better selection in the past. Kynar becomes embrittled and discolored after hydrazine exposure (Ref. 167), and a reduction in tensile strength occurs (approx. 19% in hydrazine and 35% in UDMH [Ref. 103]) after 30 day exposure at 122°F. These effects combined with lack of real need for this type of material result in it not being recommended for use or further investigation. While vapor exposure data are lacking, similar effects would be anticipated depending on concentration of vapor and exposure period.

Conflicting data on Kel-F exists but the data indicating a relatively high degree of incompatibility with hydrazine base propellants must exclude this material from consideration when compared to available alternate materials. The prime reason for consideration of Kel-F is its similarity to Teflon. It is made from the same molecule using a chloride rather than fluorine atom, and as such the compatibility would be expected to approach Teflon. Kel-F shows poor compatibility with MMH at temperatures up to 160°F for periods up to 4 weeks (Ref. 199). Other data from the same source indicates no excessive MMH decomposition at room temperature for up to 3 months, at +158°F Kel-F did have an excessive decomposition rate.

5.2.1 AF-E-411, AF-E-124D & Teflon

As indicated earlier, all three materials exhibit a high degree of compatibility with hydrazine base propellants. The basic initial difference is in the seals provided. Teflon exhibits the typical plastic sealing characteristics and disadvantages, with the problem areas of cold flow, flaking, and relatively low contamination tolerance. Since the fuel seal problem is much less severe than with N_2O_4 , the inert nature of Teflon is less advantageous. For these reasons, Teflon is not recommended as a prime candidate for fuel system seals.

The comparison between AF- E-411 and AF-5-124D can be based on differences in goals for developing these materials and performance. AF-E-411 was developed specifically as a hydrazine base propellant system seal material, while AF-E-124D was developed specifically for

 N_2O_4 systems. The performance of both materials is similar with the exception of compression set. AF-E-411 exhibits lower compression set characteristics both in air and propellant and those characteristics recommend it as an optimum material. The only justification for use of AF-E-124D in a fuel system would be the need for identical materials in both oxidizer and fuel systems. Since this is a logistics/manufacturing problem, other selection criteria would be required.. The long term characteristics of AF-E-411 (and AF-E-411A) are very good with little degradation occurring as a result of propellant exposure. There are variations in compression set and strength characteristics dependent on the propellant however. In Figure 5.4 the tensile strength retention as a function of exposure time to 5C/50 propellant is shown. Up to 11 months, this is essentially a straight line at over 100% which is 100% within experimental error. The extrapolated time for 7 days at 200°F indicates 100% retention for the long term. This is indicative of the stability of the material in 50/50. Compression set is also a constant over the long term as seen from Table 4.2-18. The maximum compression set in 50,50 is approximately 35%, up to 9 months at 75°F. This stability is also characteristic of AF-E-411A.

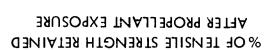
As discussed earlier, these characteristics recommend AF-E-411 as an optimum seal material for hydrazine base propellant systems.

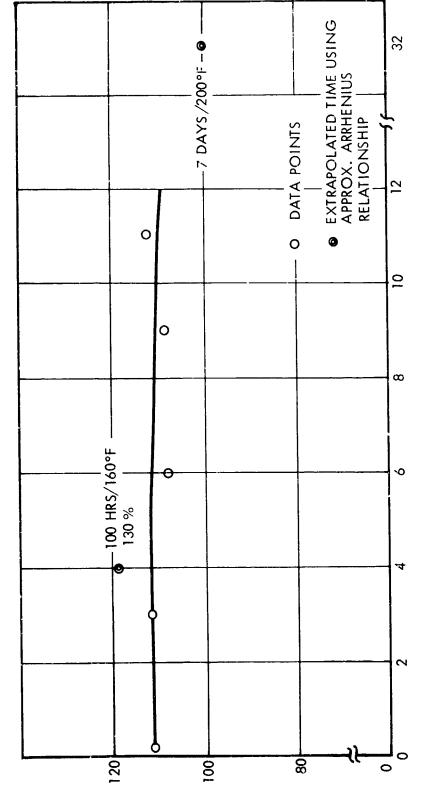
5.3 Fabrication and Availability of AF-E-124D and AF-E-411

These elastomers are typical elastomers with respect to fabrication with the exception that some of the improved properties result in characteristics which require slightly different fabrication techniques.

5.3.1 <u>AF-E-411</u>

AF-E-411 can be fabricated using common elastomer fabrication methods. The most common method is to mold to the final shape. For most designs this is relatively simple since shrinkage characteristics are well known. For complex shapes more care is required and may require a few iterations, particularly if very close final tolerances are required.





PROPELLANT EXPOSURE PERIOD (MONTHS)

AF-E-411 Tensile Strength Retention With 50/50 Fropellant Exposure Time Figure 5.4

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Other techniques using molded sheet stock may be used such as machining and grinding simple configurations. The only possible difference is more difficulty in grinding due to the high tear strength. Use of high speed, sharp grinding wheels will permit grinding after some technique development. Care must be taken not to use coolants when machining or grinding, or if coolants must be used to recognize potential incompatibility and dimensonal change due to swell.

There is presently only one source of AF-E-411, which is TRW Systems. However, facilities there are sufficient to provide any reasonable quantity and to provide molded parts. Should there be a requirement for quantities or sizes beyond TRW capability, the Air Force and TRW would work with a supplier to establish this capability.

5.3.2 AF-E-124D

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AF-E-124D also can be fabricated by conventional methods after techniques are established. The molded part can be worked in the same manner as AF-E-411. Molding of AF-E-124D is considerably more difficult than other elastomers from a number of viewpoints. In order to develop the properties characteristic of AF-E-124D, as characterized in this report and shown in the specification (Appendix E), extreme care and precision is required during the extended cure cycle. Any significant variation, can result in materials which will not meet the specification requirements. When proper care and controls are used, however, consistent quality molded parts are obtained.

The other molding problem area results from a high (\sim 12%) non-uniform shrinkage characteristic. This complicates mold design and usually requires iterations to obtain parts with correct dimensions and tolerances. Once the mola is designed correctly, uniform high quality parts are obtained. Some thick sections may have distortion due to the high shrinkage characteristic, requiring some design care.

The availability of AF-E-124D material is somewhat more limited than AF-E-411 material since the base raw stock is manufactured in batch lots. This base polymer is supplied to TRW Systems by

E. I. Du Pont de Nemours & Co., the manufacturer. The material is available in any reasonable quantities within reasonable periods of time. Should the demand significantly increase, it is expected that the source of supply would increase, and TRW would make provisions to increase the supply of molded parts or assist another source in satisfying the need.

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6.0 SEAL DESIGN PROCEDURE

This section is designed to provide basic and applied seal design information resulting in better utilization of available data and information.

In Tables 6-1 through 6-5 are listed some factors affecting non-metallic seal durability and an assessment of the most critical parameters for various non-metallic seal types. In evaluating a seal design and determining the areas of maximum emphasis these factors should be utilized and given consideration. Since each seal design and application will differ, these general assessments will not always apply but should be given initial consideration.

Before beginning the discussion of specific seal design techniques and procedures, a general discussion on polymeric material properties and behavior is presented. Realizing that a large percentage of existing designs involve either hard plastic or metal-to-metal closures, it is considered important to orient the designer toward the differences between non-polymeric materials and polymers, particularly elastomers.

6.1 Polymeric Material Properties and Behavior

The designer attempting to create or analyze seal designs must be familiar both with the generalized and specific properties and behavior of the materials from which the seals are to be made. Most designers are far more familiar with non-polymeric materials than with polymers therefore a review of the fundamentals of polymer properties and behavior is presented here as a convenience to the users of the seal design procedure which follows.

Table 6-i. Summary of Factors Affecting Non-Metallic Seal Durability

Degr	Degradation Descriptors	riptors		Pert	Pertinent Parameters	Seal	Seal Types Susceptible	ceptible		
Category	Mode	Mecasism	Geometry	Operating Conditions	Material Properties	Static	Dynamic	Poppet	di	1140
	Brittle	Impact	Volume	Kinetic energy temperature, loading rate	Impact toughness, yield strength, ductility, Modulus of elasticity			*	×	*
	Cracking	Static fatigue	Area	Load, temper- ature, time	Static fatigue endurance limit	×	*	~	×	×
	Cracking	Cyclic fatigue	Area	Load, tempera- ture	Endurance limit, Modulus of Elasticity, ultimate strengt ⁱ i	-	×	*	~	*
	Cracking	Stress-cracking	Tusckness	Load, tempera-	Load, tempera- Stress-cracking resistance ture, time, flutd	*	*	*	~	~
Fracture	Tearing	Separation		Load, loading rate, temperature	Tear strength, tear energy, energy per unit volume at rubture		×	×	×	*
	earing	Depressurization	"mickness	Magnitude and rate of ores- sure change, fluid, temperature	Permeability, ultimate tensile strengti, elongation at rupture	*	۲	-	-	
	Cutting	Shearing	Inckness, edge radius,	Load, temperature	Shear strengt:		×		~	•
	Cutting	สำห ล -draw) กฎ	Insckness	Pressure differential. fluid oroper- ties.	(Mardness or tearing energy ³)	*	•	*	*	*
	LATTUSION	7.0g.	Trackness, opening width, opening radius	Pressure differential, temperature time	Ultumate tensile strength, tensile modulus, creen modulus	×	*	×		≺
	Abrasive	Scrubbing	Sliding distance, surface finish	Load, velocity	Marchess, yield strength in shear, coefficient of friction		×	=	~	
	Adhesive	Adnesion and motion	Stroke	Load, time			*	×	-	ĸ
r e	£rosive	Impingement		otream velocity, fluid density and mass flow, temperature, time	Érosson trençi', energ, absorption coefficient		*			-
	en recorde	Chemical actaco		emperature, time, fluid corrusiver	Incrtness		•	*	~	•

Summary of Factors Affecting Non-Metallic Seal Durability (Cont.) Table 6-1.

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Degr	Degradation Descriptors	riptors		Purt	Pertinent Pira aturs		[1):5 c.	שניובמשטביי		
Categor,	Mode	Месналіsл	/rience5	Openating Conditions	Material Promortius	5.4236	7.17.8.11.0	Foppet	ני	, ta
	Swell/ Shrink	Fluid absorption/ desorption	Thickness	Flund, temper- ature, time	Solubility parameters	×	×	×	×	*
_	Expansion/ Contaction	Thermal bulk change	Thickness	Temperature, time	Coefficient of thermal exnansion, bulk compressibility, specific heat	×	*	×	×	×
Change in Size,	Blistering	Internal gas Thick generation or expansion	Thickness nsion	Temperature, time, pressure	Permeability, inertness, activation energy	×	×	×	×	
Shape, or Resili-	Blistering	Differential permeation	Thickness	Temmoerature, time	Pormeability	×	×	×	×	
?	Set	Creep and recovery	Strain	Temperature, time, fluid reactivity	Creep compliance, inertness, Modulus of Elasticity, coefficient of thermal expansion, yield strength	×	×	×	×	
	Relaxation	Stress relaxation	Strain	Temperature, time, load	Relaxation Modulus, Modulus of Elasticity	×	×	×	×	×
	Hardening	Redox reaction		Temperature, time, fluid reactivity	Inertness, permeability	×	*	×	×	×
	Hardening	Loss of plasticizer		Temperature, Iner time, fluid reactivity, pressure	Inertness, permeability ssure	×	*	×	×	×
	Hardening	Cross-linking		Temperature, Inert time, fluid reactivity, radiation	Inertness, permeability, radiation resistance assion	×	×	×	×	×
	Softening	Redox reaction		Temperature, time, fluid reactivity	Inertness, permeability	×	×	~	×	
	Softening	Depoly- merization		Temmerature, time, radiation	Inertness, resistance to thermal degradation, radiation resistance	×	*	×	×	
	Material 1055	Washing. leaching, blooming		Temperature, time, fluid reactivity	Permeability	×	×	×	×	
Mısmatcı	Misalign- ment Separation	Mechanical error Contamination	Tolerances a gimensions	Temperature	Hardness, Modulus of Elasticity	× 3	× ;	×	×	1
						Ē	×	×	~	

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(1) Secondary failure only

Table 6-2.

Summary of Critical Parameters for Non-Metallic Dynamic Seals

		Lea	akage Control		Ì		Durability	tv	
		Seal Materia	al	Design		Seal Material	ıterial	Design	
		Effec	ct on Seal Perf	t on Seal Performance if Parameter is:	meter is:	E	Effect on Seal	Performance i	Performance if Parameter is:
Parameter	Desired Property	Worst Case	Deficient But Not Worst Case	'finimum Consíderation	Not Considered	Worst Case	Deficient it Not Worst Case	Not Considered	∿inimum Corsideration
Inertness (Propellant and Space Environment Compatibility)	High	Critical	Critical	Critical	Critical	Critical	Criticai	Critical	(ritical
Contamination Sensitivity	Low	Critical	Gritical	Major	Critical	Critical	Major	Critical	Major
Permanent Set	Low	Major	Minor	Major	Critical	Critical	Major	Critical	Major
Sealing Load	Low	Major	Major.	Major	Critical	Critical	Major	Critical	Major
Material Temperature Effects	Low	Critical	Major	Major	Critical	Critical	Major	Critical	'lajor
Permeation	Low	Minor	Minor	Minor	Minor	Minor	Minor	Minor	Minor
Surface Finish (Seal)	High	Major	Major	Critical	Critical	Critical	Major	Oritical	Major
Surface Finish .(Gland or Seat)	High	N/A	N/A	Critical	Critical	Critical	Major	Critical	Major
Hardness	High	Major	Minor	Major	Major	Major	Major	Major	'ajor
Stroke (Sliding)	Low	N/A	N/A	Critical	Critical	N/A	N/A	Critical	Critical

Summary of the Most Critical Parameters for Non-Metallic Static Seals Table 6-3.

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		Leakage	eakage Control (and Durability	d Durability)	
		Seal Material	terial	Design	gn
-		Effect (on Seal Perfo	Effect on Seal Performance if Parameter is:	ter is:
Parameter	Desired Property	Worst Case	Minimum But Not Worst Case	Not Considered	Minimum Consideration
Inertness (Propellant and Space Environment Compatibility)	High	Critical	Critical	Critical	Critical
Contamination Sensitivity	Low	Critical	Critical	Critical	Critical
Permanent Set	Low	Major	Major	Critical	Major
Material Temperature Effects	Low	Major	Major	Critical	Major
Permeation	Low	Critical	Major	Critical	Major
Surface Finish (seal)	High	Major	Major	Major	Major
Surface Finish (Gland or Seat)	High	N/A	N/A	Critical	Major
Hardness	High	Major	Minor	Major	Major

Table 6-4.

Consideration ัสำกาฒนศ Critical Critical Critical Parameter is: Hajor Major Major Minor Unor Major .linor 'Innor Desian Considered Critical Critical Critical Critical Critical Critical Critical Effect on Seal Performance if lajor. tot Minor 'la,jor Major Durabilıt\ Jeficient But Not Worst Case Critical Major Major Major Major Minor 1inor Minor Minor Minor Major Seal Material Summary of the Most Critical Parameters for Mon-Metallic Poppet Seals Critical ' Critical Critical Worst Case Major Major Major Winor Minor Major Major Major Consideration "linimum Critica] Effect on Seal Performance if Parameter is: "inor Major Major 112,101 Minor Minor Minor Major Minor. Minor Design Not Considered **Crit**ical Critical Critical Major Major Major Minor 'la.jor Minor Major Major Deficient But Not Worst Case Critical Major Hinor Minor Minor Minor "inor Minor Minor Ainor N/A Control "aterial Leakage Critical Worst lajor "ajor Minor 'lajor Major Hinor Minor Minor Minor N/A Desired Property High High Hıgh High ¥0.7 <u>₹</u> Low Ľ0. MO' Low <u>۳</u> AP Sensitivity (Mt %.)
Properties, etc.) Inertness (Propellant and Space Environment Compatibility 'Material Temperature Effects Surface Finish (Gland or Seat) Surface Finish (Seal) Parameter Contamination Permanent Set Sealing Load Sensitivity Permeation Hardness Mismatch

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Table 6-5.

		Summary of t Leakage	f the Most Cr age Control	itical Paramet	Summary of the Most Critical Parameters for Non-Metallic Ball Valve Seals Leakage Control	allic Ball	Valve Seals	s Durability	
		Seal M	l Material	٥	Design	Se	eal Material	Design	ign
		Effect (ct on Seal Pe	on Seal Performance if Parameter is	arameter is:	Effect	t on Seal Performance	rformance if Parameter	ameter is:
Parameter	Desired Property	Morst Case	<pre>Peficient But Not Worst Case</pre>	Not Considered	Minimum Consideration	Morst Case	Deficient But Not Worst Case	Minumum Consideration	^ot Considered
Inertness (Propellant and Space Environment) Compatibility)	High	Critical	Critical	Critical	Critical	Critical	Critical	Critical	Critical
Contamination Sensitivity	LOW	Critical	Major	Critical	Critical	Critical	Critical	Critical	Critical
Permanent Set	רסיי	Major	Major	Major	Major	Major	Major	Critical	Critical
Sealing Load	Low	Major	Major	Major	Maj	Major	Major	Critical	Critical
Material Temo_rature Effects	Low	I Major	Major	Critical	Major	Major	Major	Critical	Critical
<pre>LP Sensitivity (Mechanical Properties, etc.)</pre>	Low	Critical	Critical	Critical	Major	Major	Major	Critical	Critical
Permeation	Low	Minor	Minor	Minor	Minor	Minor	Minor	Minor	winor
Surface Finish (Seal)	High	Critical	Major	Critical	Major	Major	Major	Major	(ritical
Surface Finish (G ¹ and or Seat)	High	N/A i	. A/N	. Critical	Major	Critical	Critical	(ritica)	Critical
Hardness	Hign	Major	Major	Major	Major	Major	Major	Major	'lajor
Stroke (Sliding)	Low	N/A	N/A	Critical	Major	Major	Major	Critica`	Sritical

Factors Determining Polymeric Properties

Polymeric material properties are largely determined by four factors:

- 1) molecular weight
- 2) structural organization
- 3) compounding
- 4) state

Molecular weight is usually stated as an average and perhaps would be more meaningfully expressed as a molecular weight distribution or spectrum. Pure polymeric substance tends to be comprised of a statistical distribution of molecular weights because neither chemically nor in the processing is there any absolute control mechanism to govern the number of monomer units in a polymeric molecule (Reference 38). Hence, most production processes result in mixtures of molecular weights. Fractionation may be used as a post-polymerization step in narrowing the range of molecular weights in a batch. Taking as an illustrative example tetrafluorethylene (Teflon), at room temperature the low molecular weight fraction is oily, the intermediate weight fraction is waxy, and the high weight fraction is solid. By mixing these in different proportions, materials of different properties are obtained.

Polymeric materials are often categorized according to structural organization in order to generalize about their mechanical properties. If poly are are visualized as long chains of atoms then at least three characteristic arrangements may be recognized: (1) amorphous-random orientation of the chains, (2) crosslinked - with lateral chain-to-chain connections, and (3) crystalline - highly ordered arrangement of the chains.

Using again the example of Teflon, Du Pont scates (Reference 29) that the TFE resin they provide for extrusion, moldings, and castings of film yields a crystallimity ranging from 50 to 70 percent when the processor follows their directions.

Figure 6.1 shows how typical property curves would compare for a polymer which can exist in varying degrees of molecular orderliness (Reference 38).

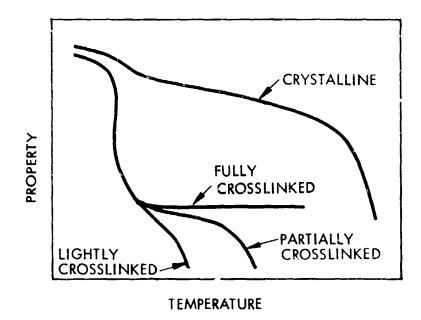


Figure 6.1. Variation in Property-Temperature Curve with Degree of Molecular Orderliness

Voids are those volumes within the bulk of the finished part which are not filled with polymer. Void content is controlled during processing. Specific gravity, strength, permeability, and dielectric strength are some of the properties which change with void content in a pure polymeric substance.

A given body of material often contains both amorphous and crystalline regions; the extent of these regions can change both due to thermal history and to load history. Crosslinking is a chemical process so the maximum amount of crosslinking will depend on the presence of suitable activators (sulfur in many rubber systems) or active side groups along each chain which can join chains together. Crosslinking can be decreased either chemically or thermally.

Within amorphous regions the chains are relatively free to move relative to each other, hence at a given temperature the bulk behavior of the material is more like a fluid. Properties tend to be isotropic (equal-valued in all directions).

Development of crosslinks tend to make the material stronger, more elastic, and stiffer partially because energy can be stored in the crosslinks. Very often crosslinked materials are more susceptible to chemical attack because the crosslink bonds are more easily broken by oxidizers or reducing agents than are the bonds in the chains which constitute the only structure in amorphous material.

Working and temperature cycling may cause amorphous regions to become crystalline. The percentage fraction (by mass) of the total bulk which is ordered in the crystalline state is labelled the "crystallinity" of the polymer. Highly crystalline materials are rigid, may be brittle, and are sometimes non-isotropic (properties vary with direction). Crystalline regions suddenly revert to the amorphous state when the temperature reaches the melting temperature $T_{\rm m}$; under certain conditions a discontinuity occurs in the volume change at $T_{\rm m}$ (Reference 38).

Controlling crystallinity is one means available to the processor for controlling properties without changing the chemical constituency of the product.

Polymeric materials may be used as either relatively pure substances or as polymeric compounds. Pure substances are comprised of only one molecular composition; for seals these are typified by Teflon (tetrafluoro-ethylene), Kel-F, polyvinyls, polyethylenes, etc. Pure substances seldom have all of the optimum mechanical property values desired for seals, for which reason they are often mixed and processed with other ingredients to produce a polymeric compound. Compounds, as mentioned herein, are mechanical mixtures of ingredients which develop some cohesiveness through crosslinking during a curing process.

Polymeric compounds generally consist of a base polymer, activators, fillers, wetting agents, plasticizers, anti-oxidants, etc. The base polymer chains are crosslinked together under the influence of the activators which either furnish the connecting link to which the side groups become attached (e.g., sulfur in vulcanized rubber), or remove terminal atoms or groups from the side groups to ready a site for crosslinking or behave like catalysts.

Fillers are either inactive except for some surface wetting or attached by crosslinks to the polymer; their function is to occupy space and provide strength, abrasion resistance or hardness (Reference 22). Wetting agents cause the fillers to be wetted so they can be firmly anchored in place (i.e., resist separation and washing). Plasticizers are effectively molecular lubricants which reduce internal friction, making the compound more flexible and capable of flow (during forming) and less prone to heating when flexed. Anti-oxidants give the compound resistance to oxidation.

Properties of compounds are very dependent upon the ingredients and processing. Degree of crosslinking, fraction as filler, size and shape of filler particles and fraction as plasticizer are especially important in determining mechanical properties. After curing, compounds are still susceptible to changes in properties if the chemical/thermal environment is not compatible with maintenance of the compound's integrity. Crosslinking can be increased (which hardens the material), or crosslinks can be washed out (reducing its strength, hardness, and abrasion resistance). And plasticizers, which are especially mobile, can be leached out or bloom (causing the material to become stiff and perhaps cracked).

The properties of polymeric materials vary considerably with state. Temperature is the state parameter which is most significant. One source states that ". . . glass transition temperature is perhaps the most important characteristic parameter of an amorphous polymer" (Reference 38). The glass transition temperature, $T_{\rm g}$, is a specific temperature (or narrow temperature band) at which most properties change radically, and below which the properties are "glassy". In qualitative terms, over a wide temperature range, amorphous polymers display a variety of characteristic tendencies ranging from "glassy" to liquid. Figure 6.2 illustrates these qualitative properties.

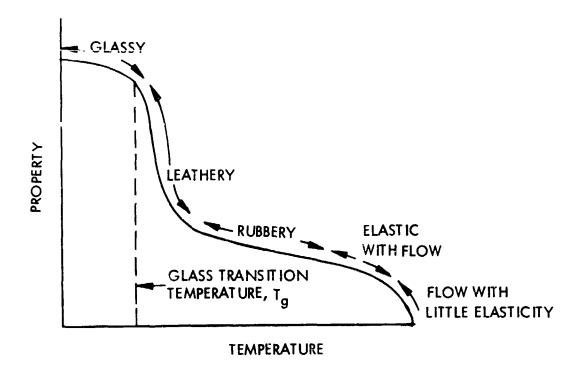


Figure 6.2. Variation in Amorphous Polymer Characteristics

The mechanical properties of polymers are generally functions of temperature and time. A widely-accepted relation for the time-temperature dependence of amorphous rubbery materials at temperatures above the glass transition temperature is that of Williams-Landel-Ferry (WLF) (Reference 21):

log
$$a_T = -\frac{C_1^0 \ (T-T_0)}{C_2^0 + T-T_0}$$
 where: $a_T = \text{magnitude of shift}$ along log time scale $C_1^0 = \text{constant at reference}$ temperature

 $T = \text{temperature for Which}$ property is being calculated

 $T_0 = \text{reference temperature}$
 $C_2^0 = C_2^0 + T-T_0$
 $C_2^0 + T-T_0$
 C_2

This equation may be used with reference to the glass transition temperature in order to make use of the fact that the constants C_1 and C_2 are nearly "universal" at that temperature:

(

()

This WLF relation is used to construct "master curves" from empirical data giving properties as time/temperature dependent functions through the process of superposition. To do this, the time coordinate (abscissa) is set up as log time and the temperature dependence of the data is factored out by shifting the plot along the abscissa. Figure 6.3 represents the basic process but the original article should be consulted (Reference 39) before this is attempted. Once these master curves are constructed the property can be predicted as a function of time and temperature by the reverse process. The WLF equation apparently holds over the range $T_{\rm g} < T < T_{\rm g} + 216 {\rm ^oF}.$

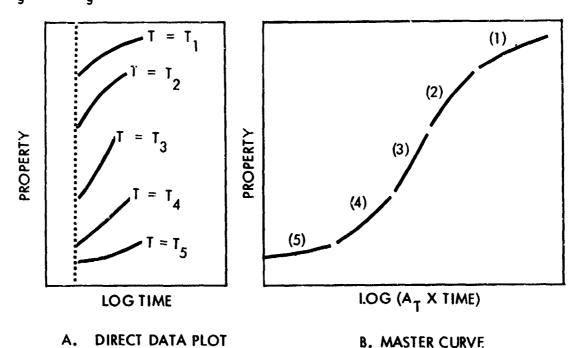


Figure 6.3. Construction of "Master Curves"

Integrity of the Polymeric Material

Inertness of polymeric materials to the fluids they will come in contact with is essential for predictable mechanical properties and durability.

In practice thermo-chemical degradation is often classified in terms describing the changes in the physical appearance of the polymer. Some polymers exhibit no thermal degradation except molecular weight changes ("unzipping" of the chain) and/or sublimation; Teflon is an example of this type of material. Other polymers tend to be made brittle due to loss of plasticizer, to incur cracking and finally to melt. Highly crosslinked polymers may soften at high temperatures if the bonds in the links are thermally-degradable.

More often than not a reactive chemical species is in contact with the polymer which is then subject to chemical attack or combined thermal plus chemical degradation. Chemical attack is usually focussed on the crosslinks either breaking them or increasing their number ("over-curing"). Drying or increased tackiness is commonly seen as the first symptoms of chemical attack. Environmental stress cracking may occur. Severe degradation causes the polymer to become either hard or soft. Hardness is most often a sign of over-cure (too many crosslinks). Breaking of crosslinks may cause the polymer to revert to gum or a crumbly mass with little cohesion. Softening sometimes is due to breaking of the polymer chain.

The propensity for one surface to stick adhesively to another is termed "Tack". Methods of measuring Tack are outlined in Reference 48. Tack may be a normal property of the surface of a material, or it may occur or be increased due either to a reaction with the fluid or the adsorption of a layer of foreign substance on its surface. Tack often increases with temperature increase. Tack is generally an undesirable characteristic in seals.

Since the leakage rate in the parametric model is directly proportional to hardness, it is essential that the seal not become too much harder than when installed. (In this context the meaning is change in hardness

at a given temperature, not change due to reduction in temperature.)

Polymer hardness is a chemically-related property in most cases, but there are certain exceptions such as when working or thermal cycling causes crystallinity to increase. Strain hardening is a common occurrence. Increases in molecular weight can occur under the right circumstances such as irradiation, chemical environment, etc. Crosslinking is another frequently-occurring cause of hardening; it is most likely to happen in compounds, highly permeable materials, and molecular structures with residual side groups which are not fully reacted. Loss of plasticizer also leads to hardening, and since most plasticizers are merely dissolved in the structure rather than being chemically-attached, they are very prone to blooming and being leached or washed out.

Because so many mechanisms cause hardening, and the chemistry is infinitely varied, there are no general relations for prediction of hardening (Ref. 21).

Reduction in hardness (softening) is not so clearly detrimental as increase in hardness, nevertheless there usually is a practical minimum. This minimum hardness is a necessary property limit mainly because extrusion and wear must be avoided. For static seal applications where there is 100% material entrapment, leak-tightness has been maintained even after seals had lost all cohesion or reverted to gum. However these extreme cases of hardness loss are of little more than curiosity value in assessing a practical minimum hardness for general application.

Softening may be caused by most of the same mechanisms that cause hardening, except the effect is reversed. There are few work-softening polymers. Molecular weight decreases (scissions) can be caused by irradiation, chemical attack on the bonds in the molecular chain, and by thermal decomposition/depolymerization. Most redox reactions attack bonds in the crosslinks; in the extreme case so many may be broken that the material reverts back to uncured state (base polymer plus compounding ingredients). Solvents cause softening if they are able to swell the material. Plasticizers in excess tend to bloom but the retained fraction will soften the material to a certain extent. Washing or leaching out of fillers usually will cause softening.

Inertness with respect to unstable propellants may not be limited to redox interactions but in certain circumstances may include catalytic activity. An example is the catalysis of hydrazine decomposition by carbon black, a common filler in rubber. To a limited degree the inertness of a material/fluid combination can be predicted from chemical activation energies but generally inertness is determined by tests.

Occasionally there is a reaction at the interface between the seal and the adjacent parts. These reactions usually result from release of a constituent of the polymer or a fluid being desorbed from the polymer. Apparently catalysis can play a role in some of these reactions. As example, rubber parts sometimes cause localized attack when held against aluminum.

Change in size (as well as change in properties) can be caused by loss of material. Change in size due to loss of mass almost certainly will lead to loss of sealing stress (and hence lead to increased leakage) unless the seal is spring loaded. Mass loss at the surface may also roughen the surface.

Except for wear, in most cases the loss is limited to fillers, plasticizers, residual activators, wetting agents, etc., which can be washed or leached out of a compound due to the lack of crosslinking with these ingredients. Blooming occurs due to simple migration of free material from the interior to the surface of the part.

To avoid changes in properties or size, and to avoid introduction of contamination into the fluid system, the resistance of seal materials to this migration is an important property. Migration mechanisms may be divided into two categories, those which are self-activating and those which are caused by external agents. In the former category are such actions as outgassing and evaporation. In the latter, wear, washing and leaching.

Outgassing normally is the result of trapped or generated gas being released through the combined mechanisms of diffusion and desorption. Vaporization of constituents within the polymer, especially in compounds, is common where either low pressures or high temperatures are imposed on the seal. Exposure to very high vacuum, as in space, will cause the

constituents with any appreciable vapor pressure to boil-off. Light ends of plasticizers, mold release agents, wetting agents and cleaning solvents are likely vapors. Trapped air may be released. Generated gases which are the products of redox reactions may also be released.

The relative immunity of a polymeric material to roughening and abrasive wear of its surface is called its "abrasion resistance" (Ref. 47). Fillers for elastomers are often chose for their enhancement of abrasion resistance. Abrasion resistance is determined by scratch or grinding tests in which the weight or volume of material removed is the principle measurable. High abrasion resistance is desirable in seals.

Washing and leaching result from soluble substances being carried from the polymer in solution. Washing removes materials near the surface, leaching transports the dissolved substance from within the bulk of the polymeric part.

Thermal Properties Especially Pertinent in Seal Design

The coefficient of thermal expansion is very large for polymers compared to most non-polymeric structural materials. Hence thermal stresses and misalignments are likely to occur unless the seal designs provide compensating features.

Thermal conductivity is an important factor in determining the susceptibility of a body of polymeric material to heat build-up degradation, thermal shock, thermal ratcheting, and thermal fatigue. It is neither an easy parameter to measure accurately nor one for which there is any simple theoretical expression useful in predicting its value in polymers.

The range of thermal conductivity values measured at room temperature is about 10 to 1 for the types of polymers used in seals. For a given material, the thermal conductivity value may vary more than 10 to 1 over the temperature range of LH₂ boiling point to +400°F. Hence, between two materials at the opposite temperature extremes there may be a difference of 100 to 1 or more in the thermal conductivity.

Mechanical Properties and Behavior

The "Hardness" of a material is not a uniquely valued property but instead depends on the method of measurement. Most of the Hardness measurement methods consist of determing how deep an indentation is produced when a specially-shaped indentor is pressed under a fixed load (force) into the surface being tested. The Shore Durometer is a special indenter with a built-in readout dial which is widely used for gauging the Hardness of polymers. Shore A Durometer is the commonly used Hardness scale for soft rubbers and plastics. Harder polymers are tested on the Shore D Durometer scale. Ver hard polymers may be rated on the Rockwell R scale. Universal correlations of hardness with other properties do not seem to exist.

Poisson's Ratio, μ , is most simply defined as the ratio of the lateral strain to the strain along the load axis; the sign of the lateral strain is the opposite from that of the axial strain (Ref. 40). This definition applies only for strains up to the proportional limit.

Poisson's Ratio varies from 0.05 or less (Beryllium) to 0.5 in soft rubber. Most metals have a Poisson's Ratio between 0.25 and 0.35. Reference 43 lists some calculated values for Teflon as 0.46 for TFE and 0.48 for FEP. Reference 44 suggests that in the step-wise solution of certain plastic strain problems, two different Poisson's Ratios might be used if μ is less than 0.5 - one for the elastic component and one for the plastic strain.

An interesting point is that for a value of about 0.5, a material does not change volume under uniaxial load, whereas for all other values of μ it does. Materials like rubber with μ = 0.5 behave like a fluid in that pressures exerted in one direction are transmitted equally in all directions. This is important in seal design since it means that pressure loading is very effective in generating sealing stress (Ref. 29).

Bulk Compressibility (or Bulk Modulus or Modulus of Compressibility) must be taken into account when calculating total strains in thin sections if the stress level is a significant fraction of the Bulk Compressibility.

Bulk Compressibility, k, is defined as the ratio of the uniform triaxial (hydrostatic) stress to the volumetric strain. For most polymeric materials which are essentially isotropic (Ref. 18) k is directly proportional to the modulus of elasticity.

In elastomeric seal design work the most valuable mechanical properties data are curves of stress versus strain as a function of strain rate, time under load, and temperature. These curves are especially valuable since polymers exhibit strong tendencies to undergo creep, stress relaxation, and set.

Many elastomers exhibit little direct proportionality between stress and strain so the modulus of elasticity must be identified in terms of the point along the curve where it was calculated. Strain per unit length is usually called Elongation in elastomers, so the 100 Percent Modulus is the stress at 100 percent elongation and the 200 Percent Modulus is the stress at 200 percent elongation.

Polymeric parts subject to fast loading and unloading cycles may respond with an apparent stiffness, or lag in strain response to stress (Ref. 13). This is due to the fact that only a portion of the energy is stored in the part as elastic deformation and the remainder is expended in overcoming viscous resistance; the latter energy component generates heat. Taking the simplest model, consider a viscoelastic part subject to a sinusoidal loading. Stresses within the part will be the resultant of two components, an elastic component which is in phase with the strain and an viscous component which leads the elastic component by 90 degrees. The viscous component must be 90 degrees in advance of the elastic component because the viscous resistance is proportional to the rate of strain, not to the instantaneous magnitude of strain. It can be shown that the resultant of the two stress components (or the total applied load) is a sinusoidal stress which leads the elastic component (or strain) by the phase angle δ ,

where:
$$\delta$$
 = arctangent $\begin{cases} \frac{\text{amplitude of viscous stress}}{\text{amplitude of elastic stress}} \end{cases}$

Tangent δ is called the loss factor.

To characterize viscoelastic behavior, a parameter has been defined which is called the "Dynamic Modulus", $E_{\rm D}$, which is calculated from the sinusoidal stresses and strains:

$$E_D = \frac{\sigma_m}{\varepsilon_m} \frac{(\sigma)_{elastic} + (\sigma)_{viscous}}{\varepsilon_m}$$

Because the elastic component is recoverable, the modulus representing that portion of the stress is called the "Storage Modulus", where:

$$E_{s} = \frac{\sigma_{melastic}}{\varepsilon_{m}}$$

Dynamic response of viscoelastic materials is discussed at length in References 12 and 36.

Members whose function is (at least in part) to absorb energy by deformation must have a suitably high "specific damping energy". Materials with high damping dissipate energy as heat due to their internal friction (hysteresis). Damping, $D_{\rm H}$, an empirically measured quantity is related to stress and temperature as (Reference 14):

 D_H = $Be^{qT}\sigma^n$ Where: B = constant, depends on material e = 2.718 q = exponent, depends on material T = temperature σ = completely-reversed stress amplitude n = exponent, depends on material

Strains in stressed parts cend to be diminished when the stress level is decreased. Perfectly elastic parts would have unique value of strain for every value of stress (i.e., all strain energy would be instantly recoverable). Real parts, however, undergo internal energy dissipation processes which take time and are functions of temperature and the strain history. Certain metals, ceramics, and rubbers may exhibit very fast recovery. "Resiliency" is generally taken to mean the fraction of the strain energy which can be immediately recovered; the re-bound height of a rubber

ball dropped on a hard surface, for example. "Resilience" is a term used to describe the amount of strain energy stored in the elastic range. Resilience is important in the design of valve seat seals because it determines the energy contributed by the seal to "bounce-back" upon rapid closure, and to opening the valve. Resilience is proportional to the elastic strain energy per unit volume up to the proportional limit (Reference 14), or,

$$U_p = \frac{\sigma_p^2}{2E}$$
, where: $U_p = \text{Modulus of Resilience, in-Lb/in}^3$

$$\sigma_p = \text{Stress at proportional limit}$$

$$E = \text{Young's Modulus}$$

A member strained into the plastic region releases more energy when unloaded than if it is taken only to the proportional limit; this action is called hyperelastic resilience, U.

Characteristic properties of a material measured at the point of failure are strongly influenced by the temperature, strain rate, and structural state. Generally materials become stronger (rupture at higher stresses) and more brittle as the temperature decreases but the relation is not linear. Also, the measured strength increases and the elongation decreases for very rapid strain rates. Materials which are crystalline tend to be stronger but elongate less than those which are amorphous.

A fully brittle material absorbs all impact energy in elastic deformation up to the point where it shatters. A fully plastic material, on the other hand, dissipates all impact energy in plastic deformation up to the point where rupture occurs. (A single material can display both of these characteristic responses, although not necessarily having the same areas under the stress-strain curves, if the rate of loading is varied; an example is "silly putty" which will shatter under impact but plastically flow under a steady load.) Most polymeric materials respond with some elastic and some plastic strain when impacted, that is, they are neither fully brittle nor fully plastic.

Also called "Tear Resistance", the Tear Strength is the load per inch of thickness required to propagate a cut or notch. Tear Strength is usually specified with relation to a specific specimen shape (e.g., nicked crescent). High Tear Strength is very desirable a most seel configurations.

The friction properties of polymers are functions of load, rubbing speed, temperature, and surface conditions. Most of the reported friction coefficients of seal materials are for "unlubricated" surfaces but that condition is not precisely defined in most cases. Generally it means that no substance was deliberately applied to the surface with the intention of lubricating it. However, tests have shown that the friction coefficients of certain material combinations, especially metal, are much higher than normal if the unoxidized substrate is exposed and the rubbing occurs in a vacuum, which indicates that the normally occurring surface films and fluid media act as lubricants. Besides oxidation layers most materials have adhering films of various substances such as mold release agents and adsorbed gases. Usually the natural lubricating properties of "dirty" surfaces are insufficient to achieve really low coefficients of friction, hence the common use of well-adhering, high-lubricity substances such as hydrocarbons and molybdenum disulfide on rubbing surfaces. These lubricants often are not compatible with rocket propellants or are less than adequately permanent (most are susceptible to being washed away or evaporated).

A few materials are sufficently self-lubricating to serve as good bearing materials. Among these are Teflon and some of the polyimides. Such polymers sacrifice a layer of material from their rubbed surface to form a lubrication film on the opposing surface. As long as this film remains intact the coefficient of friction stays very low even in a vacuum. In these cases there usually is an optimum range of surface finishes for the harder of the two rubbing surfaces which is just rough enough to abrade away a sufficient amount of the softer material to form a good lubrication film; surfaces which are substantially smoother than the optimum fail to generate a good lubrication film so the coefficient of friction is actually higher than for the optimumally-rough surface.

A variety of fluids can act as lubricants including some of the rocket propellants. This has resulted in the common observation that when valves are actuated while wet with a lubricating fluid they require less force and show reduced wear compared to when they are actuated while dry. The differences in "wet" versus "dry" wear rates has been so great in some cases that valve cycle life is often specified with a quota of dry cycles which is far less than the allowed number of wet cycles.

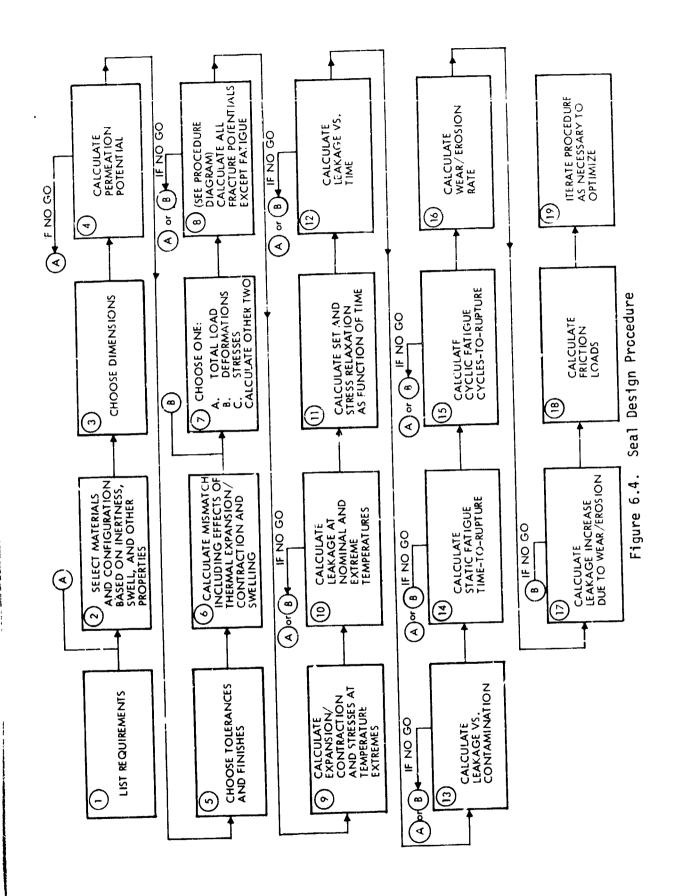
6.2 <u>Seal Design Procedure</u>

This section outlines a seal design procedure which was developed with the following philosophical points in mind:

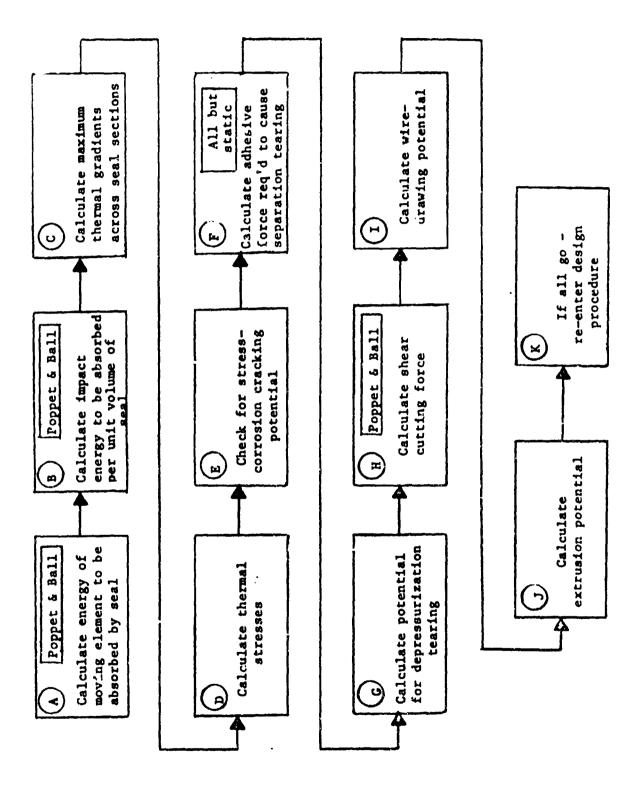
- 1. Eventually it should be possible to analytically design seals when all the necessary relations have been developed and all the necessary input data are available.
- In order to identify all the needed relations and data, it is necessary to define the overall calculating framework; only then is the logic apparent and the presently lacking data pinpointed.
- 3. Until all relations and data become available, use of the presently workable parts of the procedure minimizes the amount of "cut-and-try" effort needed to develop a seal.

6.2.1 Procedure

Figure 6.4 is a block diagram showing all the basic steps in the procedure. Individual blocks may signify very complex calculations or a lengthy iteration process. As an example, Figure 6.5 illustrates the steps in calculating the fracture potential (Step 8 in overall procedure). Figure 6.4 has also been included at the end of this section as a foldout for reference while using the design steps.



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Figure 6.5. Fracture Potential Calculation Procedure Diagram

Calculation Make/Don't Make Criteria

The designer cannot blindly follow the calculation routine. Whenever possible, short "make/don't make" criteria should be employed to judge the criticality of a parameter or design property so effort is not expended on calculations of answers not essential to success of the seal design.

Inputs

Inputs fall into the general categories of operating conditions, material properties, service life requirements, and performance goals. It may be difficult to assemble a complete set of material properties data. Such a compilation would include stress-strain, creep, relaxation, recovery, permeability, wear factors, swell, etc., as a function of loading, temperature, chemical environment; a comprehensive set of data is seldom available.

Calculation Method

This procedure in its entirety can be used today but certain steps within it are beyond our present technological resources. Those steps which meet the "make" criteria and can be implemented should not be neglected or bypassed if available input data are sufficient to carry out the calculations. Exceptions are those cases where a full warranty of success is given by complete (and reliable) empirical data. In the detailed description of this procedure on the following pages, presently available calculation methods are shown; hopefully all of these steps can be organized so as to be suitable for computer programming.

Go/No Go Criteria

Go/no go criteria are needed for many of the steps so results may be assessed before going on to the following steps. These criteria should include intrinsic limits (e.g., ultimate material properties), design goals, yardsticks of good design practice, etc.

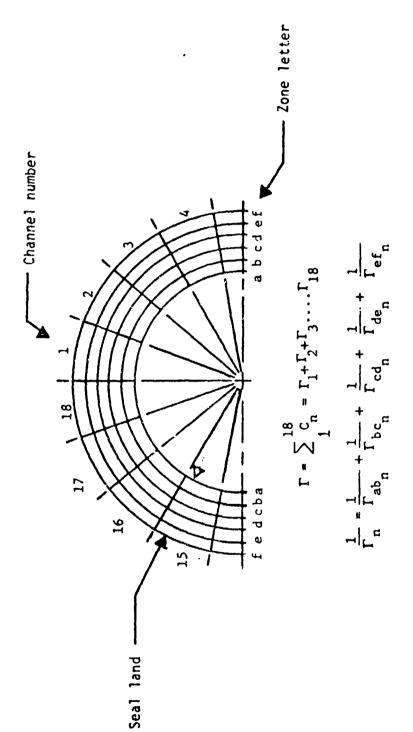
6.2.2 Leakage Model

The leakage model is based on the concept that there is a mass conductance parameter, r, which is computable and equal to the mass leak rate L divided by a flow potential term, B: (Reference 2)

$$L = \frac{B}{\Gamma}$$

The simplest case is when circumferential and radial symmetry is assumed (i.e., there are no variations in stress level or properties or finish). In that case a lumped expression for conductance can be used. But if it is desired to account for local variations, as from flange distortions, real stress distributions, etc., then a summation process must be used. As an example, consider a circular flat land across which leakage flows. Summations may be made by dividing the land into a number of radial flow channels to account for circumferential variations; the overall conductance is the sum of these parallel conductances. For radial variations, these channels can be divided into concentric radial zones; the conductance for one channel is the sum of this series of conductances. Figure 6.6 is a diagram showing the general case of radial channels comprised of concentric zones. The formulas for summing tnese parallel and series conductances are shown also. A relatively complex procedure for computing the conductance characteristic of a surface using a square grid has been developed by General Electric. (Reference 9).

One advantage of the conductance summation process is that it leads to a possible way of analytically predicting the effects of local features such as flaws and contamination particles. Figure 6.7 illustrates one approach to modelling the presence of a particle on the hard surface which has been enclosed by a soft seal material. It is assumed that the contour of the softer material and the bearing stress under it can be modelled (see below) and that the range of influence is limited (there is a line of seal-to-seat contact around the particle and another line outside of which essentially full sealing stress exists). (Ref. 88). Hence in those channels and zones covered by the range of influence, the conductance will be different than over the rest of the sealing surface.

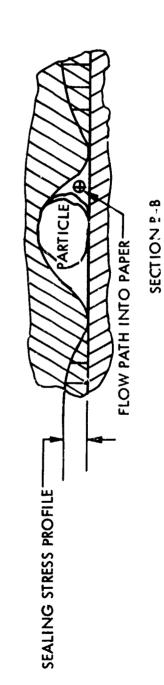


Approach to Generalized Conductance Mapping for Leakage Prediction Figure 6.6.

SECTION A-A

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Extenstion of Conduction Mapping to Cover Contamination Sensitivity Surface flaws such as cracks would be handled in a similar manner NOTE: Figure 6.7

To calculate the altered conductance piecemeal requires a model of the particle, the flow cross-section adjacent to it and the bearing stress contour between the line of contact and the one of full sealing stress. The latter two problems are the most difficult to solve. Apparently there are only two ways presently available to solve these problems:

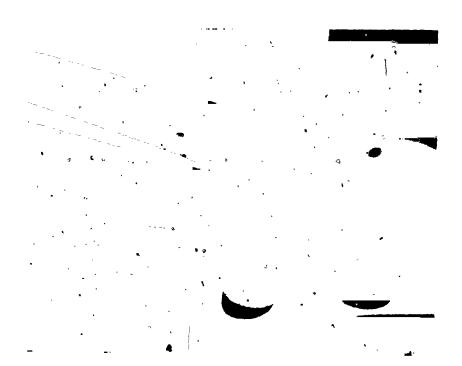
1) measure them with scale-models or 2) use a finite-element computer analysis. Although these particlar problems have not been solved, TRW has used its finite-element program to determine the stresses and strains in an elastomeric seal. (The same job involved predicting the propagation of a flaw, i.e., a void or cut, in the seal to the point of failure.) An analyst has given the opinion that, with suitable extensions to handle changes in properties with temperature, this program could be used to calculate the contour over a particle (or crack) and the adjacent bearing stresses. However, this process is time consuming (and fairly expensive).

One method of emperically determining seal deformation of complex shapes without the use of a finite-element program is through the use of two dimensional models as shown in Figures 6.8 and 6.9. This technique involves using a fixture which will retain a scale elastomer cross-section and the mating surfaces. The seal is then deformed progressively, using the cross hatch lines to determine approximate deformation patterns. A complex seal shape is shown in Figure 6.8 and in Figure 6.9 is shown the deformation of a typical O-ring cross-section.

6.2.3 Choice of Configuration

Choosing the seal configuration(s) is, of course, one of the most critical steps in any seal development. It is chiefly in the selection of the seal configuration (and any adjustments to it proven necessary by the design calculation results) that the designer exercises judgement and brings to bear his creative skills. That judgement is largely derived from experience and familiarity with state-of-the art materials and seal designs. Hence a written guide cannot be a substitute for judgment and experience.

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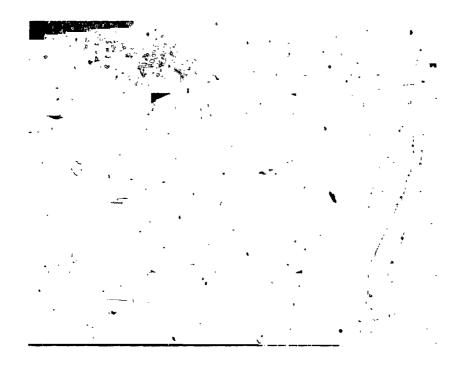


Fully Compressed

Figure 6.8 Deformation Pattern of Complex Seal



Uncompressed



Fully Compressed

Figure 6.9 Deformation Pattern of Typical O-Ring

Nevertheless, there are certain guidelines and generalized axioms which help even the experienced designer to remember some key points. Such points could be summarized in a "check list" of things to consider while selecting configurations for trial calculation. A complete listing is beyond the scope of this report but some of the pertinent subjects are briefly reviewed below.

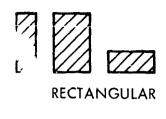
To properly engineer a seal configuration, sufficient consideration must be given to the overall installation which encompasses as a very minimum three members:

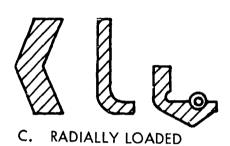
- 1. Carrier or mount
- 2. Seat or mating surface
- 3. Seal proper

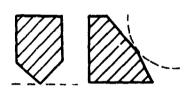
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Major considerations with regard to the carrier or mount are limitations on how the seal may be installed/removed, how the actuation force is transmitted to the seal; how the seal is to be fastened to the carrier; what provision may be made to control misalignment; extrusion clearance; and the degree of exposure of the seal to fluid pressure. For certain types of static seals which deform like hydrostatic fluids under pressure (e.g., rubber 0-rings), the void volume in the gland may be critical as may be the cross-sectional shape of the gland. If the void volume is too small, swelling or thermal expansion may cause the seal to extrude or to force the gland faces apart. Perpendicular gland walls or those with reentrant angles allow less extrusion than glands with substantially entrant walls. With regard to the seat or mating surface the prime factors are the surface finish and the sealing surface angle and width limits.

Seal cross-sections are their most individual characteristic. A wide variety of cross-sectional designs have been tried but a very high percentage of them are of configurations which fit into one of the six fundamental forms shown in Figure 6.10. In addition there are complex seals built up of several elements. These include redundant seals, spring loaded seals, seals with backup rings, etc.







E. LINE CONTACT



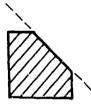


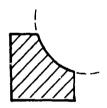
B. CIRCULAR (SHOWING SIMPLE AND COMPLEX FORMS)





D. CUP OR U-SHAPE





F. BEVELED AND SPHERICAL LAND

Figure 6.10 The Six Fundamental Forms of Seal Cross-Section Configurations

Shapes are chosen under the influence of a large number of considerations, among which are:

- 1) Achieving sufficient sealing stress
- 2) Geometric stability potential
- 3) Keeping friction low
- 4) Minimizing wear
- 5) Avoidance of problems caused by creep
- 6) Preventing extrusion
- 7) Compensating for changes which lead to seal degradation
- 8) Fabrication
- 9) Installation, inspection and maintenance, removal

Seal Loading and Stresses

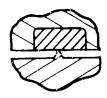
In order to generate the stresses necessary to produce sealing, loads must be applied to seals. Seal loads may be characterized by the magnitude, direction, and point of application of the forces. The internal stresses generated are a result of the interaction of these for is, the seal material properties, the environmental conditions, and the seal configuration. Seal loads originate from:

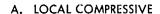
- 1. Actuator forces
- 2. Spring forces
- 3. Fluid pressure
- 4. Friction forces
- 5. Bulk behavior (such as thermal stress and elastic stress as in 0-rings stretched over a rod)

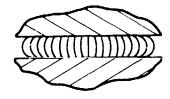
Apparently there are at least five basic mechanical loading patterns:

- 1. Local compressive
- 2. Distributed compressive
- 3. Bending
- 4. Shearing
- 5. Fully confined

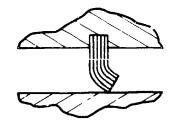
These are illustrated in Figure 6.11.



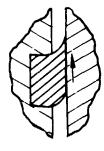




B. DISTRIBUTED COMPRESSIVE



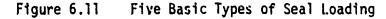
C. BENDING



D. SHEARING



E. FULLY CONFINED

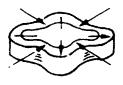


Few seals are subject to loadings purely of one category but instead most loads are combinations of two or more modes.

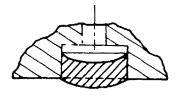
Fluid pressure forces may be directed so as to be in-line with the interface, or at some angle. Some soft polymers, especially rubbers, act as hydrostatic fluids so that the angle of the surface to which the pressure is applied makes little difference since the pressure is transmitted through the bulk and appears as normal pressure at the sealing interface.

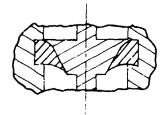
Geometric Instabilities

Instability of a configuration under certain loading conlitions can result in serious problems. Recognition of the possible forms of instability during the design phase presents the opportunity to introduce design solutions which either eliminate or at least minimize the effects of the potential instability. Figure 6.12 shows four common types of geometric instability which can cause problems in polymeric seals.









A. BUCKLING

B. ROLLING

C. DISHING

D. WEDGING

Figure 6.12 Four Types of Geometric Instability Encountered in Polymeric Seals

Buckling and dishing are usually caused by fluid pressure. Rolling occurs in dynamic (reciprocating) seals when the drag (friction force) exceeds the torsional strength of the seal. A similar result in the peripheral direction, called "wrap-around" occurs in rotary seals. Wedging results from overtravel of the moving parts which contact at an angle.

Friction Forces

In general, at a given sealing stress, friction may be kept as low as possible by proper choice of:

- 1 Material combination
- 2. Surface finish, neither too rough nor too smooth
- 3. Rubbing speeds
- 4. Areas of contact as low as feasible

Wear

Wear is a function of essentially the same parameters as those given for friction above plus the length of stroke or rubbing distance. It is serious if the seal looses sufficient material to change its shape or loading, or contaminates the system, or becomes roughened beyond allowable limits.

Creep Problems

Certain specific forms of creep can lead to serious problems, hence these potential creep situations should be recognized in the design phase so design solutions can be incorporated. Figure 6.13 illustrated four of these forms. Bulk shape changes cause changes in the stress at a given position or changes the position at a fixed total load. Micro-scale asperity interlocking causes an adhesive condition to develop between the contacting surfaces. Feather edging creates the potential for particulate contamination. Extrusion can cause cutting and interlocking of opposite parts by wedging action.

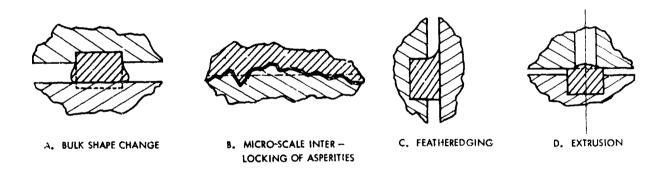


Figure 6.13. Seal Problems Caused by Creep

Extrusion

Design for prevention of extrusion involves loads, material properties and configurations. It should be remembered that surprisingly high loads can be generated within a seal by thermal expansion or swell so that even seals with adequately low extrusion clearances for the mechanical and fluid pressure loads may be extruded under these influences if the gland becomes completely filled. The solution to this problem is to provide an adequate void volume in the gland into which the seal can expand. The proportions of this channel must be judiciously chosen, however, so that at low temperature with the seal contracted it is still loaded sufficiently to seal and supported adequately against fluid pressure force to avoid gross distortion.

Compensation

Seals may be designed with features or configurations which compensate in full or in part for some of the factors which might otherwise contribute to degradation of seal performance or integrity. Some of the factors for which compensation may be provided are:

- 1. Wear
- 2. Volume change (thermal or swell)
- 3. Misalignment
- 4. Fluid pressure-load variations
- 5. Shape change due to configurational instability
- Hardness change

Fabrication

Design to achieve precision and/or reproducibility in seal fabrication must take cognizance of the type of fabrication processes to be used and the basic properties of the materials. For example, molded parts undergo shrinkage, must have sufficient relief for removal from the mold, etc. Machined parts of unstable materials (i.e., soft polymers) are difficult to finish to very smooth surface roughness and to very thin sections unless very specialized techniques are used.

Installation, inspection and Maintenance, Removal

The most common oversight in designing non-elastomeric seals is the set they take if stretched over gland parts during installation, hence the need for a complex mount which can be disassembled.

Summary of Major Design Considerations

Above all, the seal designer should keep in mind the following major considerations when choosing a seal configuration.

- Thermal cycling exerts a strong influence on changes in seal performance.
 High temperature periods induce set and relaxation which drastically
 impair sealing ability if followed by low temperature periods during
 which the seal contracts appreciably.
- 2. Soft parts must be supported so the seal can be loaded without excess creep being allowed. Distortion may occur in closure seals when valves are partially open if appreciable ΔP 's load unsupported sections of the seals. In the worst case this distortion can be so bad that the seal is damaged through gross mismatch when the valve is closed.
- 3. Excessive swell can also cause such gross changes in size or shape that the resultant mismatch either changes the valve discharge coefficient in the fully-open position or damages the seal upon closure.
- 4. Seal configuration is inextricably interconnected with seal wear and distortion patterns. Especially serious is the tendency for many configurations to result in creeping and wearing to a feather edge which eventually disintegrates into particular contamination. Also critical in pressure force balancing is consistency in the effective sealing diameter; creep and wear may change the diameter so force balances are upset.
- 5. If liquids are contained by dynamic or valve seat seals, the wear rate may be radically altered by the lubricity of the liquid. Limitations on dry cycling during test and checkout may be necessary to reach the expected in-service life.
- 6. The large differences in thermal expansion coefficients between the metal parts and the polymeric seal materials can lead to distortions and internal thermal stresses or reductions in sealing stresses unless the configuration is intrinsically immune or compensating design features are provided.

6.2.4 Design Procedure

The following pages of this section define in detail, the procedural steps shown earlier in Figure 6.4. Each step has the basic form of a table with the following information:

- Make/Don't Make Criteria (should this calculation be made?)
- Inputs Required (what information is required?)
- Available Calculation Methods (what calculation methods can be used?)
- Go/No Go Criteria (should an iteration be made or continue to the next step?)

The numbers in parenthesis in the design procedures are reference numbers and apply to the references in the bibliography, Appendix D.

The procedure diagram Fig. 6.4 has been included as a foldout at the end of this section for convenience and numbered Fig. 6.14.

STEP 1 - LIST REQUIREMENTS

MAKE/DON'T MAKE CRITERIA:

Make in all cases

Inputs Required:

- A. Environment during operation and storage:
 - 1. Temperature, fluid and external ambient
 - 2. External loading
 - a) fluid pressure, both static and dynamic
 - b) structural forces, static and dynamic
 - 3. Chemical species present
 - 4. Contamination
 - a) particulate
 - b) deposited (residues, gels, etc.)
 - 5. Vibration and shock
 - 6. Radiation
- B. Life cycle:
 - 1. Shelf life
 - 2. Duty cycle (number of cycles, length of open and closed periods, etc.)
 - 3. Post-operative
- C. Allowable leak rates:
 - 1. Duty cycle
 - 2. Post-operative
- D. Others

Available methods:

(not applicable)

Go/No Go criteria:

(not applicable)

STEP 2-- SELECT MATERIALS AND CONFIGURATION BASED ON INERTNESS, SWELL, AND OTHER PROPERTIES

MAKE/DON'T MAKE CRITERIA:

Make in all cases.

Inputs Required:

C

()

()

Materials properties data:

- 1. Inertness
 - a) Swell or shrinkage
 - b) Washout of ingredients
 - c) Propellant decomposition
 - d) Change in mechanical properties
- 2. Hardness
- 3. Permeability
- 4. Creep and relaxation properties
- 5. Thermal expansion coefficient
- 6. Tear strength
- 7. Wear resistance
- 8. Fabrication characteristics
 - a) Smoothest finish available
 - b) Tolerances on dimensions

Available methods:

Quick-check design calculations or judgements can be made based on experience. When necessary the following guidelines can be followed with regard to properties and the problems discussed in Section 6.2.3 taken into account in the configuration.

Property	Desired Characteristics
Swel1	Low as possible
Shrinkage	Low as possible
Washout	Low as possible
Propellant decomposition	Low as possible
Change in mechanical properties	Low as possible
Hardness	Best range is 70 to 80 Shore A

STEP 2 - SELECT MATERIALS AND CONFIGURATION BASED ON INERTNESS, SWELL, AND OTHER PROPERTIES

Permeability

Low as possible if low leakage desired and material is strong enough to withstand depressuri-

zation tearing

Creep and relaxation

Low as possible

Thermal expansion coefficient

Best is same as adjacent parts, otherwise as low as possible

Tear strength

High as possible and above 100

pli if possible

Wear resistance

High as possible

Finish available

Smooth as possible but at least

 $125 \mu \text{ in rms}$

Minimum attainable tolerance

on dimensions

Small as possible but at least + 0.010 in on small parts (1/2

inch diameter)

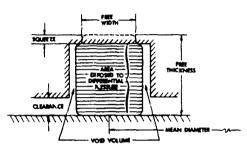
MAKE/DON'T MAKE CRITERIA:

Make in all cases.

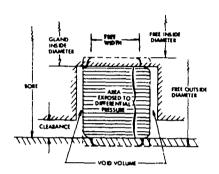
Inputs Required:

(

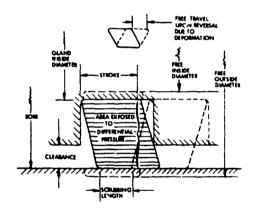
Dimensions of seal/seat assembly. As a minimum, the dimensions shown below must be fixed. For other configurations, determine critical dimensions and select values.



A. FLANGE STATIC SEAL

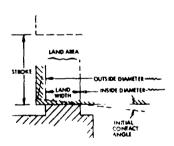


B. ROD STATIC SEAL (SIMILAR DIMENSIONS USED IN BORE SEALS)

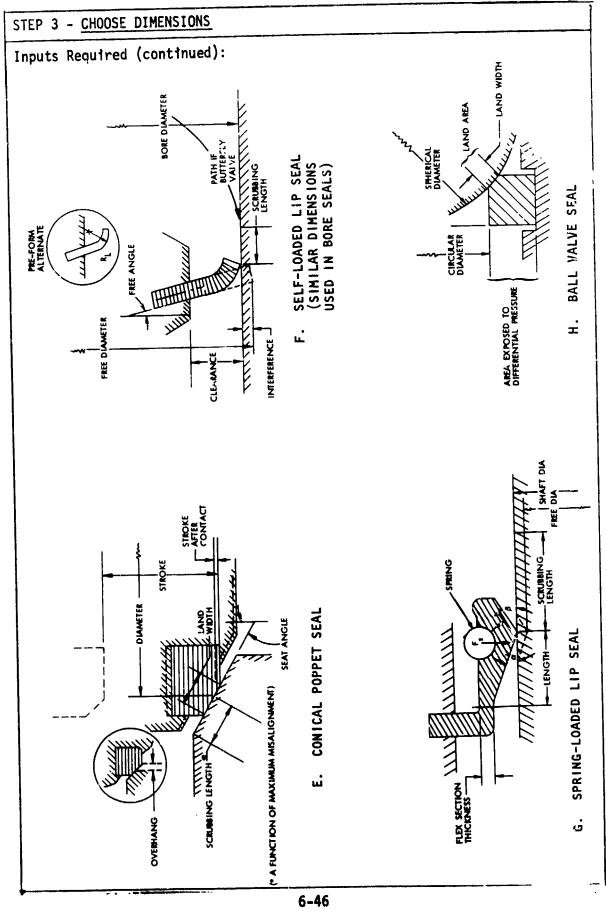


(* NOTE SCRUBBING LENGTH IS SHORTER THAN STROKE BY TOTAL AMOUNT OF FREE TRAVEL AT SCRUBBEN SURFACE)

C. DYNAMIC PISTON SEAL (SIMILAR DIMENSIONS USED FOR CYLINDER SEALS)



D. FLAT POPPET SEAL



Avaliable methods:

Short-cut design calculations and "rules of thumb" can be used to estimate reasonable dimenions. The particular calculations or rules are specific to each type of seal. Typically the following are calculated:

A. Static seals

(See References 217 for 0-ring data and 218 for flat gasket data)

1. Squeeze = free height (or diameter) - groove height free height

Typically squeezes are 0.10 to 0.30 at room temperature; squeeze at temperature extremes should be checked before selecting the room temperature-squeeze. For flat gasket seals see Reference 218 for conservative design data. For 0-rings, see Reference 217; note that the squeeze is based on the reduced diameter of the 0-ring (see below).

2. Void area = groove cross-section area - seal cross-section area.

Typically void areas should be based on the cross section of the seal at its absolute maximum temperature (not maximum operating) after maximum swelling (see step 6); a 30 percent void area at room temperature is often selected for rubber 0-rings. For 0-rings, the void area is:

 $A_v = \text{groove cross section area} - \frac{\pi}{4}w^2 (1 - x)^2$ where,

$$x = \frac{W}{10} \sqrt{\frac{6(A - K)}{K}}$$
 in glands

$$x = \frac{w}{10} \sqrt{\frac{6(B - K)}{B}}$$
 over rods

and x =amount of 0-ring diameter reduction due to stretching

w = cross section diameter of 0-ring

A = gland diameter

B = rod diameter

K = inside diameter of 0-ring

Available methods (continued)

Extrusion clearance = maximum diametral clearance seen by seal on low pressure side.

See Reference 217 for 0-ring clearances. Generally these are conservative (i.e., smaller than necessary) for other types of static seals.

B. Dynamic Seals

Dynamic seal dimensions may be chosen initially on some of the same bases as static seals and lip seals.

- 1. Interference is usually based on the allowable friction force and wear: To minimize the friction while providing adequate strength dynamic seals often are beveled to ride on an edge, hence determining the load is a complex matter. If spring loading is used, the interference is usually zero without the spring. Self-loaded seals which must seal well may have a diametral interference sufficient to generate radial stresses up to 100 psi or inner fiber stresses up to 0.1 of the yield (whichever is less).
- 2. Void area (see static seals, above).
- 3. Contact width varies over a very wide range. Choose to achieve the loadings mentioned above under "interference".

C. Poppet Seals

1. Seat Angle

Seat angles usually range from 45 to 90 degrees. Smaller angles increase wear but improve self-centering action. Sometimes the seal face angle is slightly different than that of the seat so a progressive contact is made during closure.

2. Load Width

Load width should be wide enough to achieve desired stress levels or deflections under the loads present; typically these are 50 to 500 psi for TFE (the higher values being for glass-filled Teflon) and 10 percent deflection for rubber. One source recommends

Available methods (continued)

40 to 50 percent of room temperature yield stress if the valve is to be operated at that temperature and 70 to 80 percent of room temperature yield if it is to be used in cryogenic service. (References 71 and 87).

If very large contamination particles are expected the land width should be not less than four times the largest expected particle dimension.

3. Diameter

The seal diameter is usually determined by the trade-off between the pressure (energy) loss and force balance. Pressure loss is a function of stroke, inlet/exit flow path configuration, flow velocity, etc.; a larger seal diameter compensates for a short stroke or vice versa. The force balance involves the seat diameter, poppet shaft seal diameter, the spring force (if an and the fluid pressure distribution. Usually the seal diameter is made nearly the same diameter as the shaft seal in larger valves so as to minimize the unbalanced pressure force.

4. Stroke After Contact

The amount of stroke after contact determines the degree of seal deflection, hence it must be based on allowable stress in terms of sealing and cumulative yielding and set; see 2 above.

D. Lip Seals (Reference 55)

1. Find dimensions (see Figure g, page 6-46) which satisfy the equation (Reference 222)

0.6 =
$$E\left[\frac{3}{4}S \frac{h_L}{D^2} + \frac{1}{8}\left(\frac{S}{h_L}\right)^3\right] \left(D - D_S\right) + F_R$$
, where

E = modulus of elasticity

S = flex section thickness

Available methods (continued)

 $h_1 = length$

D = shaft diameter

D_c = seal free diameter

 F_R = spring radial force per unit circumference

2. Contact (land) width should be less than 0.01 inch.

E. Ball Valve Seals (Reference 87)

- 1. Sprerical diameter (diameter of ball) = 1.7 to 1.8 times bore.
- 2. Bore of seal = 0.65 to 0.67 times spherical diameter but not less than bore.
- 3. Width of land. to achieve proper stress level ($\sigma > 60$ psi for TFE Teflon).
- 4. Sphericity of ball = \pm 0.0005 inch (Reference 71).

Go/No Go Criteria:

(Not applicable)

STEP 4 - CALCULATE PERMEATION POTENTIAL

MAKE/DON'T MAKE CRITERIA:

Make if there is a critical loss rate or total accumulated downstream mass or pressure.

Inputs Required:

Criteria for allowable permeation rate or total loss or accumulation downstream or downstream pressure.

Available methods: The steady-state permeation race, m, is (Ref. 36)

 $\hat{m} = \frac{PA(p_1 - p_2)}{t}$ Where: $\hat{m} = \text{mass flow rate}$

P = perm.ability coefficient

A = flow area

 p_1 = upstream pressure

 p_2 = downstream pressure

t = membrane thickness

Over small temperature changes the Arrhenius relation can be used to calculate a permeability coefficient for temperatures other than those for which empirical data are available (Ref. 36)

$$P = p_0 e^{-\frac{E_p}{RT}}$$

Where: P = permeability at temperature T

 p_0 = permeability at base temperature

e = 2.718

Ep = activation energy

gas constant

i = absolute temperature

Note that if E_p is not known but the permeation at two different temperatures is known, then E_p can be calculated. Over large temperature ranges, the permeability does not follow the Arrhenius relation and must be determined. Fically.

STEP 4 - CALCULATE PERMEATION POTENTIAL

Available methods (continued)

If the permeating substance fills a closed downstream volume at constant temperature with gas at pressure \mathbf{p}_2 , then \mathbf{p}_2 may be approximated as a function of time using the relation:

$$p_2 = p_1 \left(1 - e^{-\frac{\theta}{\tau}}\right)$$

Where: θ = time after induction period completed

$$\tau = \frac{Vt}{ZRTPA}$$

V = closed downstream volume

Z = compressibility factor of the gas

If the permeating substance fills a very large downstream volume with liquid having a vapor pressure, then the total lost mass ΔM is approximately:

$$\Delta M = \frac{PA}{t} \left(p_1 - p_v - p_g \right) \theta \text{ if } V_{1iq} < < V$$

Where: $p_v = vapor pressure of permeant$

 p_g = pressure of gas in downstream volume

If the permeating substance fills a small volume downstream volume, which contains a permanent gas at constant temperature, with liquid having a vapor pressure, then the total lost mass, ΔM , is approximately

$$\Delta M = \int_{0}^{\theta} \hat{m} d\theta = \int_{0}^{\theta} \frac{\hat{p}A}{\hat{t}} \left(p_{1} - p_{v} - \frac{p_{o} \cdot v_{o}}{v_{o} - \frac{\Delta M}{\hat{p}}} \right) d\theta$$

Where: ΔM = liquid mass accumulated

m = liquid permeation rate

 V_{Ω} = initial downstream volume

 p_{o} = initial downstream gas pressure

p = liquid specific mass

This implicit function must be solved by successive numerical integrations.

STEP 4 - CALCULATE PERMEATION POTENTIAL

Go/No Go Criteria:

If permeation exceeds criteria, return to Step 2. Choose a material with lower permeation if feasible to do so. If not, in Step 3, reduce dimension controlling permeation area

Z

STEP 5 - CHOOSE TOLERANCES AND FINISHES

MAKE/DON'T MAKE CRITERIA:

Make in all cases

Inputs Required:

Dimensions from Step 3

Tables or graphs of tolerances vs cost for materials to be used

Available methods:

A. Tolerances

In choosing tolerances on parts there are two factors to be considered:

- 1) The actual design requirements, and
- 2) The economic and practical limitations of current fabrication technology.

Dimensional and geometric analysis is necessary to estimate the design requirements. "Rules of thumb" are available for some types of seal designs but generally considerable analysis is required to optimize the tolerances and finish choices.

Tolerances available with current fabrication technology as a function of part size, material, and cost are best summarized in graphs from reliable sources such as the Society of the Plastics Industry, Inc. A typical format is shown below (Ref. 212).

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6-54

STEP 5 - CHOOSE TOLERANCES AND FINISHES

When these detailed and and specific graphs are not available, information can be obtained on standard materials direct from manufacturing personnel. Condensed information often relates tolerances in blocks or groups of dimensions rather than as continuous functions. Table 6-6 lists examples of tolerances ordinarily held when machining stainless steel in the TRW manufacturing area.

Unstable materials present a much greater problem. The grinding of rubber parts to precision dimensions is difficult at best so liberal tolerances may be needed. Whenever possible, polymer parts should be molded to shape and dimension. ARP's, NAS drawings, AN standards and manufacturers literature on 0-rings give typical tolerances which can be held in mass produced articles (see Ref. 214, 215, 216 and 217). Generally there is a minimum tolerance (± 0.003 inch) which should not be reduced on even the smallest parts unless very high costs can be justified.

In the absence of all other sources of data the following rules of thumb should be followed for molded polymer parts:

- 1) Molded part tolerances = mold tolerances + shrinkage reproducibility.
- 2) For mold tolerances use metal machining tolerances.
- 3) For shrinkage reproducibility use $\pm 1/2\%$ of dimension.
- 4) In most cases the total tolerance should not be less than 1% of the dimension or 0.003 inch, whichever is greater, and up to 3% is more reasonable.

B. Finishes

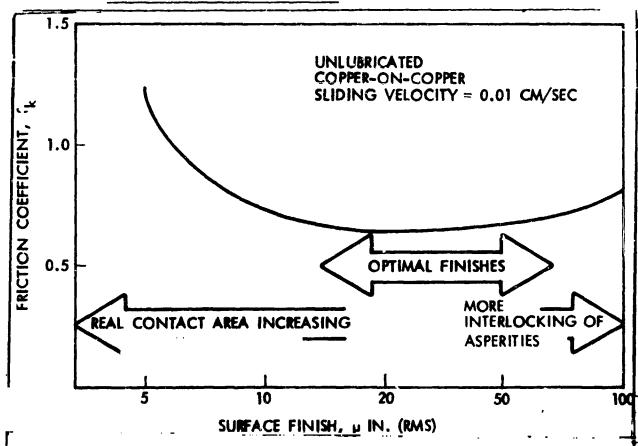
In selecting finishes it should be remembered that finishes affect:

- 1) Friction coefficient and lubrication properties.
- 2) Ease of cleaning

,		Table 6-6.	Ap	ining Precision of	proximase Machining Precision of Metal Parts of CRES
	STEP 5 - CHOOSE	TOLERANCES AND FINE	FINISHES	Available Methods (continued):	(continued): Page 3 of 6
_				Cost	
	Parameter	eter	Standard	Double	Special
-	Length	12-inch part	÷ 0.005	+ 0.002	+ 0.0001 at four times standard cost
	or	6-inch part	+ 0.002	+ 0.001	± 0.0001 at four times standard cost
. 1	Diameter	1/4-inch part	+ 0.0001	•	
.	c 1 a chance	12-inch part	+ 0.005	+ 0.002	+ 0.0092 at six times standard cost
	1 1 4 41633	1-inch part	+ 0.001	+ 0.0005	+ 0.0001 at six times standard cost
6-5	e in item to another in the second of the se	12-inch dia	+ 0.010	+ 0.005	+ 0.0005 at four times standard cost
··=	Sala La	1-inch dia.	+ 0.001	+ 0.0005	± 0.0001 at four times standard cost
• •	Roundness	1-inch dia	+ 6.0002	+ 0.0001	-
	Squareness		± 0.002 in 1 inch	± 0.0002 in 1 inch	•
	Parallelism		. ± 0.002 in 1 inch	± 0.001 in 1 inch	± 0.0002 in 1 inch at four times standard cost
1	Angle		+ 30,	+ 15,	+5' at four times standard cost
	Finish		63u-inch	32µ-inch	8µ-inch at 6 to 3 times standard cost 2µ-inch at 12 to 16 times stardard cost

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Available methods (continued)

- 3) Compatibility with propellants
- 4) Resistance to fatigue, tearing and wearing

Finish is generally called cut as the RMS* or AA* value of asperity height, wave length, and wave height. Units are micro-inches. Rough machining of metals leaves a finish of about 125μ inches RMS. Such a rough finish is never left on sealing surfaces except when large flanges are to be sealed with soft gaskets or serrations are provided. Finish machining typically is 64μ -inch but sealing areas are usually

AA = arithmetic average deviation from the mean surface

^{*} RMS = root mean square deviation from the mean surface

STEP 5 - CHOOSE TOLERANCES AND FINISHES

Available methods (continued):

specified as 32μ -inch. Finer finishes such as 8 or 16μ -inch are required for low loads, low wear rates in dynamic seals and very low leak rates with harder seal materials. Ultra-fine finishes of 2 to 4μ -inch must be made with special polishing techniques and are excessively expensive. Lapping is called for when contours must match (e.g., when a sphere-ical seat and seal must fit perfectly).

The relative cost of metal finishes is shown on Table 6-6. Carefully molded polymer parts have the same finish as the mold unless voids are left unfilled. Polymer parts may be rough if the characteristics of the polymer result in tearing during machining, however some shops have specialized techniques for grinding that leaves a very fine finish on even soft polymers.

'hen no other guidelines are available, the values listed in Table 6-7 are recommended for the initial design point.

For leakage calculations a different type of callout is needed which specifies the asperity "peak-to-valley" height, Y.** No direct correspondence exists between Y and the RMS or AA value since the shape of the asperities is involved in arriving at the RMS or AA values. If Y cannot be specified or measured, then a rule of thumb which may be used is $Y \approx 3.5$ RMS.

Go/No Go Criteria:

(not applicable)

^{**} usually the symbol h is used.

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STEP 5	_	CHOOSE	TOLERANCES	AND	FINISHES
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	Tab	le 6-7		
Specific	Surface Finish Reco	ommendations for	Initial Des	ign Point
Type of Seal	M∵terial of Seal	Desired Relative Wear and/or Leak Rate	Finish, μ-	inches RMS Seal
	Elastomer	Nomi nal	125	125
Flat Flange	Elascomer	Very low	64	64
Gasket	Plastic	Nominal	32	64
	Flastic	Very low	16	32
	Elastomer	Nominal	64	64
Static	E las coner	Very low	16-32	32
0-Ring	Plastic	Nomi na 1	32	32
	Plastic	Very low	16	32
	Floodomon	Nominai	16	32
Dynamic	Elastomer	Very low	8	32
) Dynamic	Plastic	Nomi na l	16	32
	Plastic	Very low	4-8	16
	Elastomer	Nominal	32	64
Poppet	Elesconer	Very low	16	32
Opper	Plastic	Nomi na l	16	32
	riastit	Very low	4-8	8-16
	Elastomer	Nomi na 1	32	64
Lip Seal	LIUJUMEI	Very low	16	32
.,	Plastic	Nominal	16	32
	riastic	Very low	8	16
Ball Valve	Plastic	Nomi na 1	8	32
	riastic	Very low	4	16

NOTE: For cryogenic service use lower numbers. For light gases (helium, hydrogen, etc.) use one-half the lowest number.

MAKE/DON'T MAKE CRITERIA:

Make in all cases except when high leakage rates are acceptable in seals exposed to only a narrow range of temperatures.

Inputs Required:

Criteria of acceptable mismatch

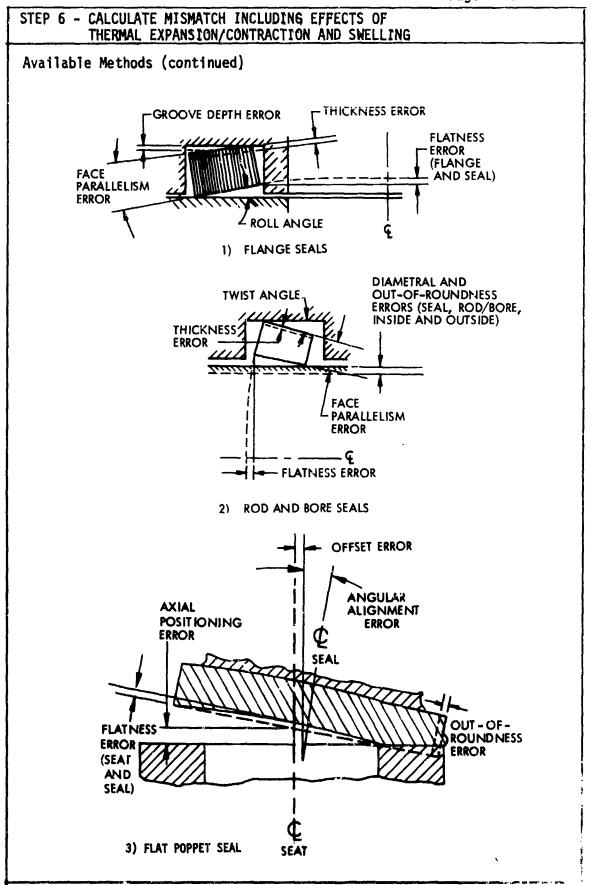
Nominal dimensions of seal and associated hardware

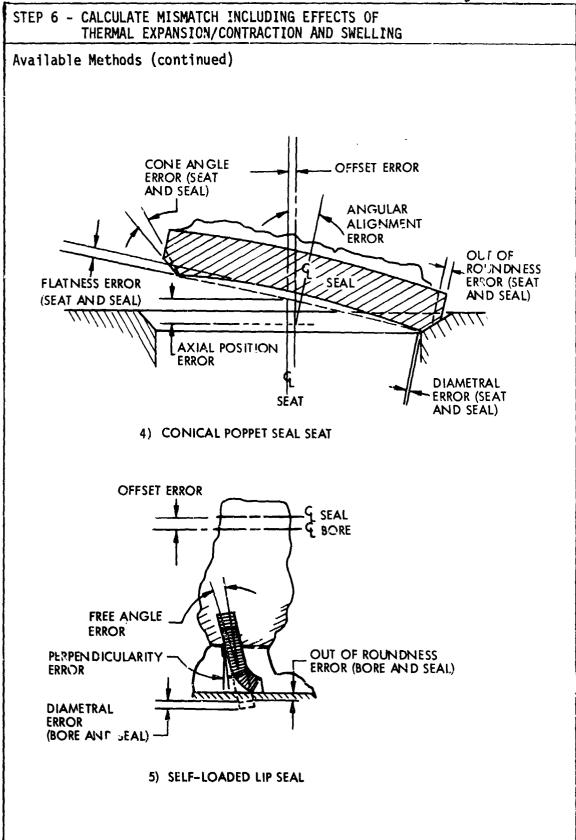
Thermal expansion/contraction properties

Swelling data

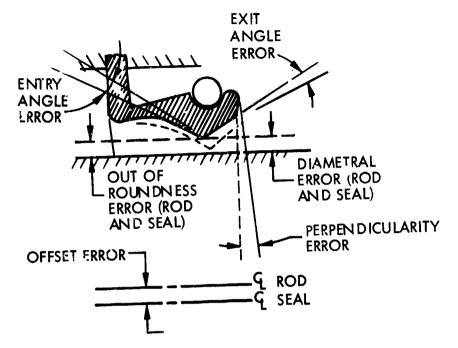
Available methods:

A. Mechanically-induced mismatches: For each design of seal there are errors which arise due to the geometrical relations and associated tolerances in the dimensions. These must be calculated for each case as no generalities can cover all the errors. The figure below illustrates the errors which give rise to mechanically induced mismatch in six kinds of seal applications. For other designs the errors should be deduced from the geometry.



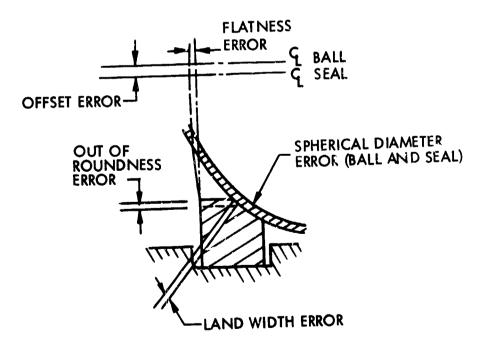


Available Methods (continued)



6) SPRING-LOADED LIP SEAL

()



7) BALL VALVE SEAL

Available Methods (continued)

B. Thermally induced mismatches:

Thermal growth in one dimension, δ_t ; due to temperature change T is:

 $\delta_t = \alpha L T$, where: α = coefficient of thermal expansion

L = dimension which changes

ΔT = temperature difference between nominal and worst case

Thermal growth in volume, ΔV :

 $V = aV\Delta T$, Where a = coefficient of volumetric expansion <math display="block">V = volume

Where empirical data are not available, a method for estimating the thermal coefficient of expansion for polymers is (38);

 $a = \frac{\gamma_C}{BV}$, Where y = constant (3.83 in CGS units) $C_V = specific heat at constant volume$ <math>B = bulk volume compressibility

C. Chemically induced mismatches:

Swell of a polymer may not be isotropic, however as a first approximation it may be assumed equal in all directions. Empirical data are generally reported as volume change, Q, which can be used to approximate the volume change, ΔV , of a fully saturated seal as:

$$V = V_0 \frac{Q}{100}$$
, Where: $V_0 = \text{seal volume with no absorted fluid}$

$$Q = \text{swell in percent}$$

Hence each linear dimension change, $\delta_{\perp},$ is:

 $\delta_L = L \frac{Q}{300}$ Where: L = dimension

Available Methods (continued):

When empirical data are not available, swell, Q, may be estimated as:

Log Q = 2.75 - 0.08
$$[(\delta_{D}-8.8)^{2} \div 0.191 (\delta_{p} + 3.9)^{2} + 0.191 (\delta_{H} - 3.4)^{2}]$$

where: Q = percent swell by volume of cc/gm

 δ_D = solubility parameter relating to dispersion forces, $(cal/cc)^{1/2}$

 $\delta_{\rm H} = \pi \omega$ lity parameter relating to hydrogen bonding, $({\rm cai/cc})^{1/2}$

The solubility parameters are:

 δ_{D} = read from graphs in reference 49

$$^{\circ}p \qquad \sqrt{\frac{2.1 \text{ KT}}{\text{VN}}} \frac{(\varepsilon - n^2)(2\varepsilon + n^2)}{(n+2)^2}$$

$$\delta_{H} = 3.8 \sqrt{\frac{\dot{\Lambda} v}{V}}$$

where: K = Boltzman's constant

T = absolute temperature

= dielectric constant

n = index of refraction for S_dium D light in Debye units

V = Molar volume

N = Avogauro's number

v = frequency in wave numbers, %/cm

Available Methods (continued):

D. Scrubbing lengths

One of the most significant outputs of the mismatch calculations are the scrubbing lengths which can be approximated with the relations $i\hat{n}$ the table. Note that the errors used are the combined effects of mechanical, thermal, and chemical influences.

Table 6-8. Approximate Maximum Scrubling Lengths

Maximum Scrubbing Lengt
s - f
™ u p ^B
e _D
e _D sin €
$\sqrt{R_{B}^{2} - R_{S}^{2} \text{ if } R_{B} > R_{S}}$ $\sqrt{R_{S}^{2} - R_{B}^{2} \text{ if } R_{S} > R_{R}}$
ПОВ

Go/No Go Criteria:

If criteria are met. go to next step. If not, return to Step 2 or 3. When no criteria are available the following rules of thumb may be used. The total effect of all summed worst case mismatches shall not:

- 1) Cause the mated surface area to be less than 80 percent of the nominal.
- 2) Cause the Jegree of seal compression to vary by a factor of more than 1.5 to 1 around any circumferential line on the mated seal surface.

STEP 7 - DETERMINE STATIC LOADS, STRESSES, AND DEFORMATIONS

MAKE/DON'T MAKE CRITERIA:

Make if low leakage or extended life is required.

Inputs Required:

Empirical data or computer program for determining stresses and large deformations in polymeric materials

Stress vs strain data on materials

Dimensions of seals

Loads

Available methods:

Choose load, stress or deformation, then calculate the other two. Finite element computer programs are required for all but the simplest cases.

As a first estimate and for inputs into leakage calculations, an "apparent stress" is calculated from the total load across the seal and the average area in contact with the mating surface. Some equations have been derived for simple cases and are presented in Table 6-9.

Bulk Compressibility

Bulk Compressibility (or Bulk Modulus or Modulus of Compressibility) must be taken into account when calculating total strains in thin sections if the stress level is a significant fraction of the Bulk Compressibility. Bulk Compressibility, k, is defined as the ratio of the uniform triaxial (hydrostatic) stress to the volumetric strain. For most materials which are essentially isotropic (Reference 18):

Page 2 of 4

STEP 7 - DETERMINE STATIC LOADS, STRESSES, AND LEFORMATIONS

 $k \approx \frac{E}{3(1-2\mu)}$ where: E = Modulus of Elasticity $\mu = Poisson's Ratio$

Pure gum rubber is stated in Reference 12 to have a Bulk Compressibility of 1.5×10^5 psi; the value increases in inverse proportion to the percent by volume of rubber in mixtures of rubber plus fillers. Reference 43 reports the calculated k of TFE Teflon is 2.07×10^5 psi and FEP Teflon is 4.14×10^5 psi. The same reference says that, due to change in E with time, k of TFE will drop 25 percent in 200 hours and 43 percent in one year; the 2.07×10^5 value is the k at 200 hours.

Reference 12 discusses the role of Bulk Compressibility in the calculation of stresses and strains within rubber parts. An example given is the case of a tnin pad of rubber loaded in compression. The total strain, ϵ_T , is:

$$\epsilon_T = \epsilon_E + \frac{\sigma}{k}$$
 where $\epsilon_E = \text{elastic strain}$
 $\sigma = \text{stress}$

Elastic strain, ε_{Γ} , is:

 ϵ_E = 1 - λ , where λ = ratio of strained to unstrained height, ignoring the correction above for k.

And λ is obtained from the implicit relation,

$$\sigma = -G \left(\lambda - \frac{1}{\lambda^2}\right)$$
, where: $G = \text{Shear Modulus} \approx \frac{E}{3}$

Reference 12 gives a table of ε_E and λ versus the term $(\lambda - \frac{1}{\lambda^2})$. If either or both of the faces of the pad are bonded, the stress, and hence ε_T , is modified by a shape factor. References 12 and 35 give shape factors for compression sandwiches bonded on both sides but no shape factors for rings bonded on one side, as in the case of seals, were located in the literature.

STEP 7 - DETERMINE STATIC LOADS, STRESSES, AND DEFORMATIONS

Table 6-9. STRESS IN RUBBER SEALS

TYPE OF SEAL	GEOMETRIC PARAMETERS	APPROXIMATE APPARENT STRESS
o-ring		$\int_{0}^{4\pi} \int_{0}^{2\pi} \int_{0}^{2\pi$
FLAT GASKET		σ* - F + P
VEE SEAL		σ* - P + E <u>Δt</u>

= A CONSTANT = LOAD PER LINEAR INCH = CROSS-SECTION DIAMETER BEFORE SQEEZE APPLIED = POISSON'S RATIO

E = MODULUS OF ELASTICITY
P = FLUID PRESSURE
W = ORIGINAL UNSTRESSEC WIDTH
t = ORIGINAL UNSTRESSEC THICKNESS

* ASSUMES POISSON'S RATIO = 0.5 SO PRESSURE IS TRANSMITTED AS IF SEAL WERE A FLUID

STEP 7 - DETERMINE STATIC LOADS, STRESSES, AND DEFORMATIONS

Go/No Go criteria:

If stresses or deformations are either too low or too high, return to Step 2 and re-design seal. Typical criteria for acceptable stresses should be compiled. Lacking this compilation, use these rules of thumb:

$$0.2\sigma_{\text{yield}} < \sigma < 0.9\sigma_{\text{ultimate}}$$
 $5\% < \epsilon < 40\%$

STEP 8 - CALCULATE ALL FRACTURE POTENTIALS EXCEPT FATIGUE

MAKE/DON'T MAKE CRITERIA:

Make in all cases except:

Omit A,B,F,&H for static seals

Omit E if fluid is inert

Omit G if pressure is less than 300 psi

Inputs Required:

Failure criteria for each mode

Dimensions, masses and velocities of parts

Thermal and mechanical properties of materials

Available methods:

A. Calculate Energy to be Absorbed

If all the kinetic energy of the moving element is to be absorbed by the seal,

$$E = \frac{MV^2}{2}$$

where: E = total kinetic energy

M = moving mass

V = velocity at instant of contact

If a bumper or stop absorbs some of the energy, then the fraction of the total absorbed by the stop, E_A , must be estimated and subtracted from the total:

$$E = \frac{MV^2}{2} - E_A$$

To obtain V, a detailed kinematic analysis is necessary in the absence of empirical data. If such an analysis is unwarranted, then an approximation may be made as follows:

Where: s = stroke

t = response time

STEP 8 - CALCULATE ALL FRACTURE POTENTIALS EXCEPT FATIGUE

Available methods (continued):

Or, alternatively:

$$E = \int (F_a - F_f - F_p) ds$$
 Where: $F_a = \text{actuator force}$
 $F_c = \text{friction force}$

 F_f = friction force

F_p = net pressure - area force (use correct sign)

s = stroke

B. Calculate Specific Energy per Unit Volume of Seal

 $S_v = \frac{E}{V_c}$ Where: $V_s = \text{volume of seal absorbing energy}$

S, should not exceed some reasonable fraction of the impact toughness of the seal material; when no other data available, use 1/3.

Determination of the actual fracture potential under rapidly applied loads is complex and involves viscoe astic and/or stress wave analysis (see References 36 and 13, respectively, for summaries).

A relative measure of the suitability of material for impact service is (14):

$$I = \frac{S_{ij}\sigma_{y}D}{\sqrt{E}}$$

= figure of merit, resistance to impact Where: I

= impact toughness

= yield stress at high strain rates

- Modulus of Elasticity

- ductility

C. Calculate Thermal Gradients Across Seal Sections

The maximum possible thermal gradient should be estimated or analytically determined by heat transfer calculations. Usually

STEP 8 - CALCULATE ALL FRACTURE POTENTIAL EXCEPT FATIGUE

Available methods (continued):

this latter course is a sizeable operation involving heat transfer network calculations.

D. Calculate Thermal Shock Potential and Thermal Stresses

Four parametric relations to be used as figures of merit for resistance of a ductile material to thermal shock and two relations for a brittle materia! are (Reference 15):

$$\theta_{1D} = \frac{D(1-\mu)}{\alpha}$$

$$\theta_{2D} = \frac{\sigma_f(1-\mu)}{E_{\alpha}}$$

$$\theta_{3D} = \frac{\sigma_f}{\sigma_{end}}$$

$$\theta_{4D} = k$$

where: D = ductility =
$$\ln \left(\frac{A_0}{A_f} \right)$$

 μ = Poisson's ratio

 α = coefficient of thermal expansion

 $\sigma_{\rm f}$ = fracture stress (true stress at rupture)

E = modulus of elasticity

σ_{end} = endurance stress

k = thermal conductivity

For brittle materials the relations are:

$$\theta_{1B} = \frac{\sigma_{b}(1-\mu)}{E\alpha}$$

where: σ_b = breaking stress (apparent stress at rupture)

STEP 8 - CALCULATE ALL FRACTURE POTENTIALS EXCEPT FATIGUE

Available methods (continued):

The above relations are useful for comparing the relative merit of candidate materials, but in some cases an absolute measure of suitability is desired so relations are needed to predict the rmal shock failure. For example, in a thin circular disc, the radial temperature gradient, $\Delta T_{\rm f}$, which will cause fracture is (Reference 16):

$$\Delta T_f = \frac{\sigma_f(1-\mu)}{E\alpha} \left[2.0 + \frac{4.3k}{ah} - 0.5e^{-\frac{16k}{ah}} \right]$$

where: $\sigma_f = \text{fracture stress}$

 μ = Poisson's ratio

E = Young's modulus

 α = coefficient of thermal expansion

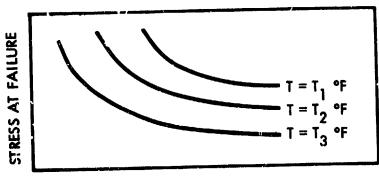
k = thermal conductivity

a = radius of disc

h = surface heat transfer coefficient

E. <u>Determine Environmental Stress-Cracking Potential</u>

Empirical data are needed to estimate the potential for environmentally induced stress-cracking. Such data are usually plotted as time-to-failure versus stress level with temperature as a parameter as shown below:



TIME TO FAILURE

STEP 8 - CALCULATE ALL FRACTURE POTENTIALS EXCEPT FATIGUE

Available methods (continued):

For computer calculations, these data must be entered either as tables or else curve-fitting techniques used to obtain the equations and constants. Note that the stress involved is the maximum surface stress, not the average bulk stress unless the empirical data specifically apply to the same configuration and load as the candidate seal and load (Reference 20):

F. Calculate Separation Tearing Potential

Generally two conditions must be met to tear a seal:

- The seal must adhere to two separating elements with sufficient peel strength to exceed the load necessary to cause tearing, and
- 2) The mechanical elements must separate under an actuation force great enough to cause tearing.

For progressive or cyclic tearing in natural rubber and some synthetics, a parameter, G, called "cut-growth constant" has been defined as (Reference !2):

$$G = T^{\times} \frac{dN}{dc}$$

where: T = tearing energy

x =empirical exponent ranging from 2 to 4

 $\frac{dN}{dc}$ = slope of curve of number of cycles versus cut depth

In some rubber compounds the "constant" G varies with rate of cycling below a limiting value (Reference 2!):

STEP 8 - CALCULATE ALL FRACTURE POTENTIAL EXCEPT FATIGUE

Available methods (continued):

The number of cycles to failure, N, is:

$$N = \frac{G}{(2KE)^{Y}c_{O}^{X}}$$

where: K = constant (which varies probably from 2 to 3)

 $c_0 = initial cut depth$

E = strain energy per unit volume

Y = empirical exponent ranging from 2 to 4

X =empirical exponent ranging from 1 to 3

More complex expressions have been obtained for N but all require empirical data to evaluate the constants and exponents. The naturally occurring material cut depths in rubbers have been found to be in the range of 1 to 3×10^{-3} cm (Reference 21):

Polymers will tear catastrophically (N = 1) as fast as the tear energy is put into the material.

G. <u>Determine The Potential for De-Pressurization Tearing</u>

At the present time, methods have not been developed to determine this failure potential. However, it has been reported (Ref. 219) that bubbles will grow in crosslinked elastomers if sudden depressurization occurs from a critical pressure p_c where:

$$p_c \ge 2.5G$$

where: G = shear modulus of the elastomer, which is about E/3 for rubber.

A more comprehensive analysis is given in Reference 220.

STEP 8 - CALCULATE ALL FRACTURE POTENTIAL EXCEPT ATTIGUE

Available methods (continued):

H. Calculate Shear Cutting Potential

Shear cutting failure will occur when the shear strength of the material is exceeded. An approximate criterion for cutting is:

$$\frac{F}{tS_s} \geq 1$$

where:

F = force p~~ unit length

t = thickness

S_c = shear strength

I. <u>Determine Wire-Drawing Potential</u>

At the present time there is no analytical method for determining the resistance of a seal to localized ensive wear which cuts a thin groove in the seal. Empirical correlations for a given gas probably would be based on the parameters ΔP , temperature.

J. Determine Extrusion Potential

The parameters governing extruction under slowly applied pressure are ultimate tensile strength, material this kness, creep modulus, tensile modulus, and the dimensions of the hole (diameter, entry radius, etc.). Generalized solutions are very complex so empirical data are needed.

Go/No Go criteria:

Compare results with failure critery, and return to Step 2 cr 1 if criteria indicates failure.

STEP 9 ~ CALCULATE EXPANSION/CONTRACTION AND THERMAL STRESSES AT TEMPERATURE EXTREMES

MAKE/DON'T MAKE CRITERIA:

Make in all cases where extreme temperatures may be reached. As a rule of thumb, make if extreme temperatures depart from the nominal by more than $50^{\circ}R$.

Inputs Required:

Dimensions of parts

Thermal stress equations in program

Thermal properties of materials

Stress vs strain and temperature data on materials

Safety margins or failure criteria

Available Methods:

Expansion/contraction of simple structural shapes can be calculated using the equations listed in Step 6.

Many thermal stress problems have been solved and solutions may be found in texts on the subject and/or by use of computer programs. Two situations give rise to thermal stresses: (1) temperature gradients and (2) two or more mutually constraining members with different coefficients of thermal expansion which undergo a temperature change from the relaxed state temperature (at which no loading occurs).

In general the induced thermal stress, $\boldsymbol{\sigma}_{T},$ is:

$$\sigma = (\alpha \Delta T \ell - \Delta \ell) E$$

where: $\alpha = \text{coefficient of thermal expansion}$

L = dimension

Δt = change in dimension actually occur. ing

STEP 9 - CALCULATE EXPANSION/CONTRACTION AND THERMAL STRESSES AT TEMPERATURE EXTREMES

Available Methods (continued):

For quick checks of elastic-range stresses induced by radial thermal gradients in ring-shaped members use the relation:

$$\frac{\sigma}{\text{Max}} = 0.577 \ \alpha \text{E}\Delta T \left(\frac{1 + \sqrt{1 - v^2}}{1 - v} \right)$$

Where: $\frac{\sigma}{\text{Max}} = \text{maximum hoop stress in outer fiber}$

 α = coefficient of thermal expansion

E = modulus of elasticity

ΔT = maximum radial temperatures gradient

ν = Poisson's ratio

A circular disc which is full constrained at its outer edge and has a temperature gradient, ΔT , induced from one face to the other develops a stress of:

$$\sigma = \frac{\alpha E \Delta T}{2 (1-v)}$$

If the temperature in the above disc is uniform but ΔT above the relaxed state temperature, the stress is:

$$\sigma = \frac{\alpha E \Delta T}{1-\alpha}$$

A bar, such as a valve stem, which is fully constrained lengthwise while undergoing a uniform temperature rise ΔT throughout will be stressed to:

$$\sigma = \alpha E \Delta T$$

The most common case in poppet valves is where the stem/poppet assembly and the seal are of different materials and/or at different temperatures than the body so thermal stresses are induced. If it may be assumed that each of these three elements is at a uniform temperature and each is uniform in cross-sectional area, then the thermally induced load change carried by the seal, ΔF , is approximately:

STEP 9 - CALCULATE EXPANSION/CONTRACTION AND THERMAL STRESSES AT TEMPERATURE EXTREMES

Available Methods (continued):

$$\Delta F = \frac{\sigma_1 L_1}{E_1 A_1} + \frac{\Delta T_1 + \alpha 2^{L_2} \Delta T_2 - \alpha_3 (L_1 + L_2) \Delta T_3}{E_2 A_2} - \frac{1}{E_3 A_3}$$

Where: subscript 1 refers to seal parameters

subscript 2 refers to stem/poppet assembly parameters

L = length of element undergoing thermal expansion or contraction

ΔT ≈ temperature difference above or below relaxed state temperature

E = modulus of elasticity at the existing temperature

A = cross-sectional area carrying load

The resulting stress increase in the seal must be calculated using the methods in Step 7.

It is important to remember that the magnitude of thermally-induced stresses and strains can be very much affected by the geometric proportions. For example, the short, thin element "x" in the sketch below can be highly stressed compared to the longer and thicker members (Reference 224).

If ΔT_{a-x} is the difference in temperature between the outer members and the center member and the cross-sectional area of x is very much smaller than that of the other members, then the thermally-induced stress (in the elastic range) is:

$$\sigma \approx \frac{L_a}{L_x} E_x = \Delta T_{a-x}$$

STEP 9 - CALCULATE EXPANSION/CONTRACTION AND THERMAL STRESSES AT TEMPERATURE EXTREMES

Go/No Go Criteria:

Compare results to margins or criteria and return to Step 2 or 7 if margins are not present or criteria not met.

STEP 1G - CALCULATE LEAKA . AT NOMINAL AND EXTREME TEMPERATURES

MAKE/DON'T MAKE CRITERIA

Make in all cases unless leakage is not of interest.

Inputs Required:

Z

Fluid conditions and properties (vicosity ratio of specific heats and gas constant)

Seal configuration with dimensions and finishes

Changes in dimensions with temperature

Stress and stress change with temperature and mismatch material hardness

Available Methods (References 1,2,3,4,5,6,7,8):

L = TB, Where: L = mass leakage rate

T = conductance

B = flow potential parameter

 $\bar{T} = \frac{CH^{X}F}{\sigma^{X}}$

Where: C = characteristic dimension and coefficients

of seal

H = hardness

F = a parameter expressing the surface finish

 σ_{s} = sealing stress

x,y = exponents

The flow potential parameter, B, must be calculated for the specific conditions of interest and the fluid properties. Table 6-9 lists expressions for B under different flow regimes.

Parameter C depends upon the configuration and the flow regime. A comprehensive table of C as a function of configuration and flow regime has not been computed. The general case for molecular flow is:

$$C = \frac{1}{\int_{-A^2}^{\frac{1}{2}} dx}$$

Where: Z = perimeter of flow path

A = cross section area of

flow path

2 = length of flow path

		Table 6-10 F	Flow Potential Parameter	m,er "B"		
Type of Mass Flow	Choked Flow	Turbulent Flow	Laminar Flow	Transition Flow	Molecular Flow	Avat
Incompressible		B = k \(\sqrt{290(P_1-P_2)}\)	Β "	;		lable me
Compressible	8 = 12 14 14 (2) 17	$8 - k \sqrt{\frac{2q}{R!}} \ln \left(\frac{P_1}{P_2}\right)$	B = k(P2-P2)	$B = \frac{k(p_1^2 - p_2^2)}{24RT_0} $ (1+A\frac{x}{h})	$B = kX(P^2 - P^2)$	theds (co
Isochermal flow witt Symbols: h = charact k = various p = upstre p = upstre p = upstre life natura Chok Turbulent flow is all transition flow is in Iransition flow is in Proper constants mus Olvide mass flow by leakage in stand Turbulent flow, adia For isothermal, turbulent flow, adia	Isochermal flow with zero approach velocity in all cases. Symbols: h = characteristic passage dimension k = various constants and discharge coefficients p = upstream pressure lh = natural logarithm To determine flow regime, check by calculation: Choked flow is for presure ratios of p₂/p₁²< (²²/ y⁻¹) Turbulent flow is all Reymold's numbers above ≈ 2000 transition to laminar is not abrupt) Laminar flow is in range of Reymold's numbers < 2000 Transition flow is in the range X/h = 0.01 to 1.0 Proper constants must be supplied to reconcile units Divide mass flow by specific mass at standard conditions to get leakage in standard volumetric units per unit time. Turbulent flow, adiabatic case for zero approach velocity is B = kp₂/for isothermal, turbulent, compressible flow with high approach velocity	in all cases. on R = specific gas respective gas temperatus R = specific gas temperatus X = man free par X = man fr	A = 5.75 for pure gase R = specific gas const R = specific gas const T = gas temperature X = mean free path Y = ratio of specific n = viscosity P = specific mass (or n = viscosity P = vis	e gases, constant in file hea if file hea in file hea in file by the file hear in file by the file by	4.21 for mixture of gases ts ts $\begin{pmatrix} A_{1} & A_{2} & A_{3} & A$	ntfnued:

STEP 10 - CALCULATE LEAKAGE AT NOMINAL AND EXTREME TEMPERATURES

Available methods (continued):

Reference 80 reports for molecular flow through a flat poppet (annulus):

$$C = \sqrt{\frac{2}{\pi}} \frac{h^2 D}{W}$$

Where: h = effective separation between

D = mean diameter of lands

W = width of lands

 $\pi = 3.14159$

Reference 1 reports for isothermal laminar flow through a flat poppet (annulus):

$$C = \frac{2\pi h^3}{\epsilon n \frac{r_0}{r_i}}$$

Where: $\pi = 3.14159$

h = effective separation between

lands

en = natural logarithm

 $r_0 = outside radius of lands$

r; = inside radius of lands

For isothermal flow through a longitudinal annulus (Reference 221) as in a rod or gland seal:

$$C = \frac{\pi D b^3 \left[1 + 1.5 \left(\frac{\varepsilon}{b}\right)^3\right]}{\epsilon}$$

Where: $\pi = 3.14159$

D = outer diameter

b = clearance (annulus

width)

ε = eccentricity

& = length of path

Hardness, H, is raised to some power which is determined empirically. For example, the exponent is 3.25 for flat gaskets of Buna-N rubber (Reference 6)

STEP 10 - CALCULATE LEAKAGE AT NOMINAL AND EXTREME TEMPERATURES

Available methods (continued):

F is the arithmetic average of the peak-to-valley (PT $^{\prime\prime}$) heights of the mating surfaces (Ref 23).

$$F = \frac{1}{S_1} \int_0^{S_1} |Y_1| dS_1 + \frac{1}{S_2} \int_0^{S_2} |Y_2| dS_2$$
,

where:

S = surface area

Y = height of asperity, peak-to-valley

Subscript 1 = seat

Subscript 2 = seal

It has been reported that vessels which have been in contact with a liquid will show no leakage below 10^{-7} scc/sec. If a slug of surface-wetting liquid enters a round passage of uniform diameter, displacement of the liquid from the massage requires a pressure differential, ΔP , of (Ref 55):

$$\Delta P = \frac{4\sigma \cos \theta}{d}$$

Where: σ = surface to tension at the liquid-gas interface

Ø = liquid-to-wall contact angle

d = diameter of passage

Go/No Go criteria: Leak rate must not exceed criteria. If it does exceed it return to Step 2 or Step 7.

MAKE/DON'T MAKE CRITERIA:

Make for any case in which seal remains loaded for extended periods. As a rule of thumb, make if seal is loaded for periods of longer than ten minutes or if the sum of all loaded periods exceeds fifty percent of the total time (50% duty cycle).

Inputs Required:

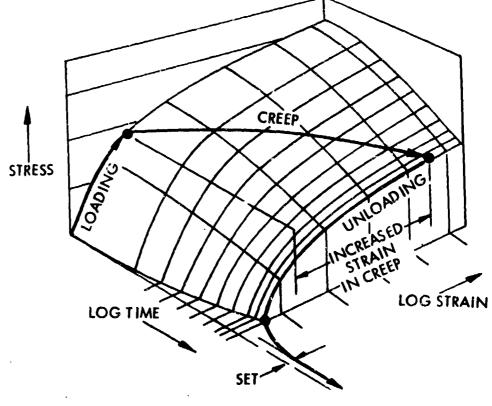
Stresses or strains in seal

Stress-strain properties of material including creep and relaxation as functions of time and temperature

Available Methods:

A. <u>Set</u>

Empirical data are needed to calculate the set which a seal will take. Stress-strain-time surfaces are required such as shown below. One such surface is needed for each temperature.



Strain vs Stress vs Time Surface, Showing Creep Path

Available Methods (continued):

To be used on computer programs the empirical data must be reduced either to tables or equations. Creep response of the material is characterized by the Creep Compliance, J(t):

$$J(t) \equiv \frac{\varepsilon(t)}{\sigma}$$
, where $\varepsilon(t)$ = strain as a function of time σ = stress

The super position method leads to tensile creep equations of the form (Reference 14 and 36):

$$\epsilon = \epsilon_e + \epsilon_p^1 + \epsilon_c^1 + \epsilon_c^3$$

where: < = total strain

 $\epsilon_{\rm D}^{+}$ = initial plastic strain

 $\epsilon_{\rm C}^{\prime}$ = transient creep strain

 $\epsilon_{C}^{"}$ = constant (minimum rate) creep strain

Which leads to:

$$\epsilon = \frac{\sigma}{E} + k_1 \sigma^p + k_2 (1 - e^{-qt}) \sigma^m + k_3 t \sigma^n$$

where: $\sigma = stress$

E = modulus of elasticity

e = 2.718

t = time

 k_1, k_2, k_3 = empirical constants

p,q,m,n, = empirical exponents

Available Methods (continued):

All of the above strains are considered to be recoverable, in time, except for the second and fourth terms. Hence, the tensile set is:

$$S = k_1 \sigma^p + k_3 t \sigma^n$$

A special mode! of creep called "thermal ratcheting" may be induced by temperature cycling if the sum of the mechanical plus the thermal stresses exceeds the yield stress. A relation for the threshold of thermal ratcheting has been developed for a structure consisting of two parallel members made of the same material, A and B, which support two parallel rigid plates (Ref. 15). Ratcheting will occur when:

$$\sigma > \sigma_y = \frac{E \delta \Delta T}{2 + A/B + B/A}$$

Where: σ = total imposed stress (load plus thermal stress)

 $\sigma_v = yield stress$

E = Modulus of Elasticity

δ = coefficient of thermal expansion

ΔT = temperature rise above or below the "zero thermal stress" temperature

A = cross-sectional area of "A"

B = cross-sectional area of "B"

For this structure, thermal ratcheting will be a maximum when A = B. The strain per thermal cycle is:

$$A\varepsilon = \delta \Delta T - \frac{\sigma_{y} - \sigma_{B}}{E} - \frac{\sigma_{y} - \sigma_{A}}{E}$$

Where: $\sigma_A, \sigma_R = \text{peak stresses in A and B}$

For zero thermal ratcheting of a pressurized cylinder in the presence of a linear thermal gradient (Ref. 13):

Available Methods (continued):

$$\sigma_{\mathbf{t}} = \frac{\sigma_{\mathbf{y}}}{\sigma_{\mathbf{p}}}$$

$$\sigma_{t} = \frac{\sigma_{y}^{2}}{\sigma_{p}}$$
 for $0 < \frac{\sigma_{p}}{\sigma_{y}} < 0.5$

$$\sigma_{\mathbf{t}} = 4\sigma_{\mathbf{y}} \left(1 - \frac{\sigma_{\mathbf{p}}}{\sigma_{\mathbf{y}}}\right)$$
 for $0.5 < \frac{\sigma_{\mathbf{p}}}{\sigma_{\mathbf{y}}} < 1.0$

$$0.5 < \frac{\sigma_p}{\sigma_y} < 1.0$$

where:

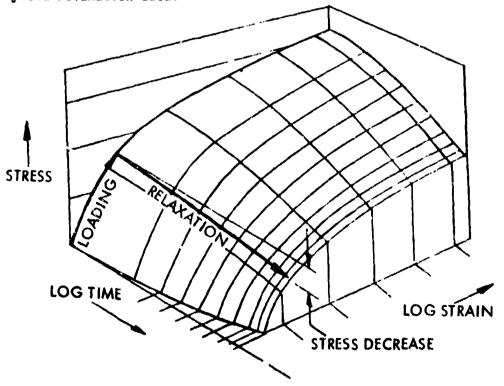
thermal stress

yield stress

pressure stress

B. Relaxation

The same empirically established surfaces used for set will yield relaxation data.



Strains vs Stress as a Function of Time, Showing Stress Relaxation

Available Methods (continued):

Relaxation behavior of a material is characterized by the Relaxation Modulus, E(t),

Where: $E(t) \equiv \frac{F(t)}{A_0 \Delta \ell / \ell_0}$, where F(t) = load as a function of time $A_0 = original$ area

AL = change in length

l_o = original length

Stress relaxation in polymers should be calculated from A relation for the short time empirical data whomever possible. stress relaxation is (Ref. 14):

$$\frac{\sigma}{\sigma_i} = \sqrt[n]{\frac{k_1}{[k_1 + k_2 (1-e^{-\eta t}) + k_3 t]}}$$

where: σ/σ_i = ratio of stress at time t to initial stress

n = empirical exponent

 k_1, k_2, k_3 = empirical constants

e = 2.718

q = empirical exponent

= time

If empirical data are available only for creep and not for stress relaxation, and these data refer to viscoelastic creep which proceeds as a homogeneous deformation of an unchanged structure, then stress relaxation can be derived from the creep data. For a simple war in tension, such as a valve stem, the transformation equation is very simple (Ref. 37):

$$\frac{d\sigma}{dt} = - E_{\varepsilon_c}^{\circ}$$

Available Methods (continued):

where: $\sigma = stress$

t = time

E = Modulus of Elasticity

0

 ε_c = creep strain rate

Go/No Go Criteria:

Not applicable

rage 1 01 /
STEP 12 - LEAKAGE VS TIME
MAKE/DON'T MAKE CRITERIA:
Make in all cases if Step 11 results indicate a change in seal- ing stress with time.
Inputs Required:
Same as Step (0)
Results of Step (1)
Available Methods:
Use method of Step (0)
Go/No Go Criteria:
Proceed to Step (13) if criteria met. If not, return to Step (2.)

STEP 13 - CALCULATE LEAKAGE VS CONTAMINATION

MAKE/DON'T MAKE CRITERIA:

Make if particulate contamination is possible.

Inputs Required:

Particle population data (size vs number).

Same as Step (10)

Available Methods:

A number of simplifying assumptions are usually necessary to make the problem solution practicable for the case of polymeric seals:

- All particles are much harder than the seal and so may be treated as non-deformable.
- 2) Particles are either uniformly distributed through the fluid, or are generated locally at a known rate.
- 3) The probability of particles being trapped on the seal can be calculated in terms of size, number and location on seal.

Based on these assumptions, calculations are made resulting in a model of the "worst case" conditions of particles on the seal, then Step 10 is repeated but with the conductance altered to account for the presence of the particles.

Alternatively, the Step 10 calculations may be made with various size and number of particles located at various places on the seal. These results are parametric only and express the "leakage sensitivity" of the seal to contamination but don't reveal what the likely leak rate increase will be.

If particle generation through seal wear is a concern, calculate the volume of wear as below in Step (16) and maximum particle size from the expression (Reference 26):

$$d = \frac{6 E W_{ab}}{v^2 \sigma_y^2}$$

STEP 13 - CALCULATE LEAKAGE VS CONTAMINATION

Available Methods (continued):

where: d = diameter

E = Modulus of Elasticity

 W_{ab} = Work of adhesion between the two surfaces

v = Poisson's Ratio

 σ_{v} = normal compressive stress

Go-No-Go Criteria:

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Same as Step (10).

STEP 14 - CALCULATE STATIC FATIGUE TIME-TO-RUPTURE

MAKE/DON'T MAKE CRITERIA:

Make if seal remains highly loaded for extended periods. As a rule of thumb, make if:

$$\left(\frac{\sigma}{\sigma}\right)^{8}$$
 times > 1, where time is in hours

Inputs Required:

Seal stresses including stress relaxation from Step (1) . Static fatigue data in materials Environment and life cycle from Step (1) Allowable margin of time

Available Methods:

Empirical data must be used to determine if the existant loads will cause fracture at the prevailing temperature before the required time period has elapsed. The required time period should include a margin beyond the actual expected time under load.

A relation which allows empirical static fatigue data at a given stress level to be extrapolated to other temperatures (13, 14, 18):

$$\log t_r = \frac{P}{T} - C$$

Where: $t_r = time to rupture, hr$

P = empirical constant

T = absolute temperature, °R

C = empirical constant (usually 20)

A similar approach can be taken with a more accurate but more cumbersome relation (same references):

$$\log t_r = \frac{T - T_a}{P} + \log t_a$$

Where: T = temperature, °F

 $T_a = empirical constant$

t_a = empirical constant

STEP 14 - CALCULATE STATIC FATIGUE TIME-TO-RUPTURE
Co/No Co Cuitouis.
Go/No Go Criteria:
If failure is predicted, return to Step 2. If not, go on to
next step.
nent step.

STEP 15 - CALCULATE CYCLIC FATIGUE CYCLES-TO-RUPTURE

MAKE/DON'I MAKE CRITERIA:

Make if high cycle life is required and there is reason to suspect fatigue may occur. As a rule of thumb, make if life cycle includes more than 10,000 cycles in the elastic stress range, more than 100 cycles in the plastic stress range, or more than 10 cycles at above 0.8 of the breaking stress.

Inputs Required:

Stress cycling and environment data from Steps 1 and 7.
Empirical fatigue data on materials

Available Methods:

Empirical fatigue data are usually plotted as "S-N" curves which sheet the number of cycles to failure as a function of stress level; to be complete these curves should have as other parameters temperature, chemical environment, etc. When hand calculations are made, these curves can be used directly or with cross-plotting to predict failure. When computerized calculations are made, either tables must be entered or the data reduced to equation form. Several of the equation forms have been determined for different types of fatigue. Extended discussions are found in References 15 and 36.

For alternating strains which are entirely within the elastic range, the cycle life $N_{\rm f}$ is (15):

$$N_f = \left(\frac{\Delta \epsilon_e E}{G}\right)^{\frac{1}{\gamma}}$$

where: $\Delta \epsilon_e = strain range per cycle$

E = elastic modulus

y = empirical exponent

G = empirical constant

STEP 15 - CALCULATE CYCLIC FATIGUE CYCLES-TO-RUPTURE

Available Methods (continued):

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In order to estimate the variation in cycle life with stress level in the elastic range, use the relation (Reference 15):

$$N_f = N_{end} \left(\frac{\sigma}{\sigma_{end}} \right)^{\frac{1}{\gamma}}$$

where: N_{end} = endurance cycle life σ = cyclic stress σ_{end} = endurance stress

A closed solution is obtained only if γ is known or if the fracture stress, $\sigma_{\bf f},$ is measured so γ can be approximated as above.

Larger amplitude strains which induce both elastic and plastic deformations involve an implicit expression for N $_f$ in terms of the total strain range, $\Delta \varepsilon_{e+p}$:

$$\Delta \epsilon_{e+p} = MN_f^Z + \frac{G}{E}N_f^Y$$

where: M = empirical constant

Z = empirical exponent

$$\gamma = -0.083 - 0.166 \log \left(\frac{\sigma_f}{\sigma_u}\right)$$

$$G = \frac{9}{4} \sigma_u \left(\frac{\sigma_f}{\sigma u} \right)^0.$$

M = 0.827 D
$$\left[1 - 82\left(\frac{\sigma_{u}}{E}\right)\left(\frac{\sigma_{f}}{\sigma_{u}}\right)^{0.179}\right]^{-1/3}$$

$$Z = -0.52 - \frac{1}{4} \log D + \frac{1}{3} \log \left[1 - 82 \left(\frac{\sigma_u}{E} \right) \left(\frac{\sigma_f}{\sigma_u} \right)^{0.179} \right]$$

where: σ_f = fracture stress

σ_{...} = ultimate stress

STEP 15 - CALCULATE CYCLIC FATIGUE CYCLES-TO-RUPTURE

Available Methods (continued):

Sometimes the cycle life is expressed as a function of only the plastic strain $\boldsymbol{\epsilon}_{n}$:

 $N_f = K \epsilon_p^X$

The following has been reported for fluorocarbons (Ref. 223):

	Tempera	ature
Material	Room	+200 ⁰ F
TFE	$\epsilon_{\rm p} N^{0.203} = 1.25$	ε _p N ^{0.433} = 2
FEP	$\varepsilon_{\rm p}^{\rm N}^{\rm 0.084} = 1$	$\epsilon_p N^{0.044} = 1$

In low-cycle fatigue, below 10,000 cycles, the plastic strain per cycle is the dominant parameter, while above that figure, elastic strains begin to be more important than below it but total strain is the most significant parameter (15). When fatigue is due to a combination of mechanical plus thermal stress cycling and the strains (or stresses) can be described by linear differential equations, the effective total strain, ε , (or stress) is simply the algebraic sum (i.e., superposition can be used) (15):

$$\varepsilon = \varepsilon_{M} + \varepsilon_{t}$$

where: ϵ_{M} = mechanically induced strains

 ε_{t} = thermally induced strains

Spalling fatigue life is markedly load dependent being inverse to the cube of the load and inverse to the ninth power of the stress (19). Experimental data are required.

Go/No Go Criteria:

If failure indicated, return to Step 2.

STEP 16 - CALCULATE WEAR/EROSIGN RATE

MAKE/DON'T MAKE CRITERIA:

Make in all cases except for seals requiring very low cycle life and/or operating at very low stress levels.

Inputs Required:

Seal dimensions and loads Wear coefficients for materials Sliding distances and velocities Allowable wear criteria

Available Methods:

Wear prediction is largely an empirical matter which is specific to the materials, surface conditions, loads and rubbing speeds.

A. Abrasive Hear

The volumetric abrasive wear rate of the safter of the two materials, V_1 , is approximately (References 19, 26, and 27):

$$V_1 = \frac{KWL}{3P_1}$$

where: K = a constant (Note: sometimes this constant is combined with the divisor 3, which is a property of the material combination.)

W = load or apparent stress

L = sliding distance

 P_1 = indentation hardness of the softer material

The harder materials' wear rate, V_2 , will be:

$$v_2 = \begin{bmatrix} \frac{p_1}{p_2} \end{bmatrix}^2 v$$

where: P₂ = indentation hardness of harder material

STEP 16 - CALCULATE WEAR/ERCSION RATE

Available Methods (continued):

To some degree the wear rate (or K) will be determined by the type of asperity deformation (elastic or plastic). A plasticity index, ψ , has been suggested.

 $\psi = \frac{E'}{H} \sqrt{\frac{\sigma}{R}}$

where: E' = an elastic constant

H = hardness

σ = standard deviation of the asperity heights

R = average radius of the asperity tips

For values of ψ below about 0.6, the deformations are largely elastic and the wear rates are low, while above 1.0, the deformations are largely plastic and the wear rates higher.

Polymers apparently experience elastic wear. PTFE moldings wear at rates which are approximately proportional to the modulus of elasticity and inversely proportional to the yield strain (Reference 26).

Use of the PV relation to estimate the wear of Teflon is recommended (Reference 30):

$$t = KPVT$$

where: t = wear, in.

K = wear factor

P = pressure, psi

V = velocity, ft/min.

T = time, hr.

For pure TFE, K is given as 2.5×10^{-7} , and for FEP as greater than 5×10^{-7} . Values of K are also given for several filled Teflon materials, and PV limits are specified.

B. Adhesive Wear

The equation for adhesive wear is the same as for abrasive but the constant, K, is different. There is a (theoretical) minimum load, L_{min} , below which adhesive wear particles will not be formed (Reference 19):

 $L_{min} = \frac{C'W'}{P}$

STEP 16 - CALCULATE WEAR/EROSION RATE

Available Methods (continued):

where: C' = a constant

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W = work of adhesion

P = penetration hardness

C. Erosive Wear

The volumetric rate of erosive wear, V is (Reference 31):

$$V = \frac{\delta I A}{S_e}$$

where: 6 = absorption coefficient

I = power of stream

A = area

 S_{p} = erosion strength

Apparently the "erosion strength", $S_{\rm e}$, is proportional to strain energy but the proportionality factor and absorption coefficient must be determined empirically.

D. Corrosive Wear

Rate equations which describe the thickness of corrosion layer, Δy , which can be built up in time, t, fall into three categories (References 26 and 28):

1. Linear,
$$\Delta y = K_1 t + K_2$$

2. Logarithmic,
$$\Delta y = K_3 \log (K_4 t + K_5)$$

3. Parabolic,
$$\Delta y^2 = K_6 t + K_7$$

where all K's are empirical constants

If the corrosion is removed abrasively by, a highly loaded point, the depth of wear per pass, h, ' approximately (Reference 19):

$$h = \frac{kx}{3}$$

where: $k = empirical constant (probably <math>10^{-4}$ to 10^{-5} on metals)

x = sliding distance

STEP 16 - CALCULATE WEAR/EROSION RATE

Available methods (continued):

E. "Zero" Wear

It may be possible to predict conditions which will lead to essentially zero abrasive/adhesive wear (Reference 33). The criterion is:

$$S_s \leq \left[\frac{2000}{N}\right]^{1/9} GY$$

where: $S_c = shear stress$

N = number of passes

G = stress concentration factor

Y = yield point in shear

Generally G is either 0.20 or 0.54 and does not appear to be a continuous function. G equals 0.20 for systems (contact surfaces and lubrication combination) with a high susceptibility of material transfer. G equal 0.54 for systems in which transfer is unlikely; for many plastics against 302 stainless steel, G = 0.54.

Shear stress, S_s , is a function of the geometry of the mating surfaces. For flat against flat, journal bearings, and balls in sockets,

$$S_s = Kq_0 \sqrt{1/4 + f^2}$$

where: K = stress concentration factor

q * bearing pressure

f = coeffi cient of friction

The stress concentration factor may be taken as 2 if all corners are well rounded. Sharp corners can lead to values of K as high as 1000. See also Reference 34.

Go/No Go Criteria:

If wear rate or total exceeds wear criteria, return to Stap 2.

rage 1 of 1
STEP 17 - CALCULATE LEAKAGE INCREASE DUE TO WEAR/EROSION
MAKE/DON'T MAKE CRITERIA:
Make if wear/erosion calculated in Step (16)
Inputs Required:
Results of Step (6)
Criteria for acceptable leakage increase due to wear/erosion.
Available Methods:
Repeat Step 6, 9, and 10 but with new inputs from Step 16.
Gc/No Go Criteria:
Return to Step(2)if criteria not met.
·

STEP 18 - CALCULATE FRICTION LOADS

MAKE/DON'T MAKE CRITERIA:

Make if actuator force output is marginal or if loads must be known accurately to predict response.

Inputs Required:

Empirical friction coefficient data for material combinations

Rubbing velocities and loads or stresses

Environment including temperature and degree of lubrication present

Criteria for acceptable friction forces

Available Methods:

Friction calculations are all based on empirical data except for a few theoretical methods which yield poor accuracy.

A relation which holds for materials such as Teflon is (References 19 and 26):

$$F = cP^{Z}$$

where: F = kinetic friction force

c = empirical constant

P = force vector magnitude, normal to surface

Z = empirical exponent, usually between 2/3 and 1

Kinetic friction for some material combinations is dependent on the sliding speed:

$$F_k = Pdv^{-y}$$

where: d = empirical constant

v = sliding speed

y = empirical exponent

Where several combinations of surfaces (1 to n) are simultanously in contact in a complex device, the total frictional force is the algebraic sum of the individual forces of the surfaces:

STEP 18 - CALCULATE FRICTION LOADS

Available Methods (continued):

$$F = F_1 + F_2 + F_3 ... F_n$$

Since the normal force may not be the same for each increment of area, the local "pressure" is used as the basis of calculation:

$$F_n = A_n C_n q_n^Z$$

 A_n = contact area for material combination n where:

 C_n = empirical constant for material combination n

 q_n = average compressive stress over area A_n

empirical exponent for material combination n

Static friction may be a function of "time of stick"; for very short times it is essentially the same as kinetic friction but increases rapidly as the time of stick increases, then, asymptotically approaches the familiar static friction value. Mathematically, the static friction force, F_s, is (Reference 19):

$$F_s = \left(f_k + kt_s^x\right)P$$
kinetic friction coefficient

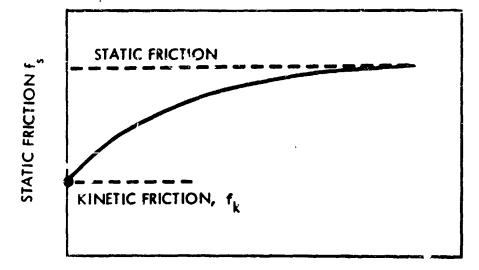
empirical constant

time of stick

empirical exponent

STEP 18 - CALCULATE FRICTION LOADS

Available Methods (continued):



TIME OF STICK

Reduced shear strength and yield strength have been cited as possible results of fluid interactions which would cause an increase in friction expressed as (Reference 36):

$$F = \left(\frac{\alpha}{\sigma y} + \frac{(1-\alpha)}{\sigma y}\right) p$$

S = shear strength of polymer

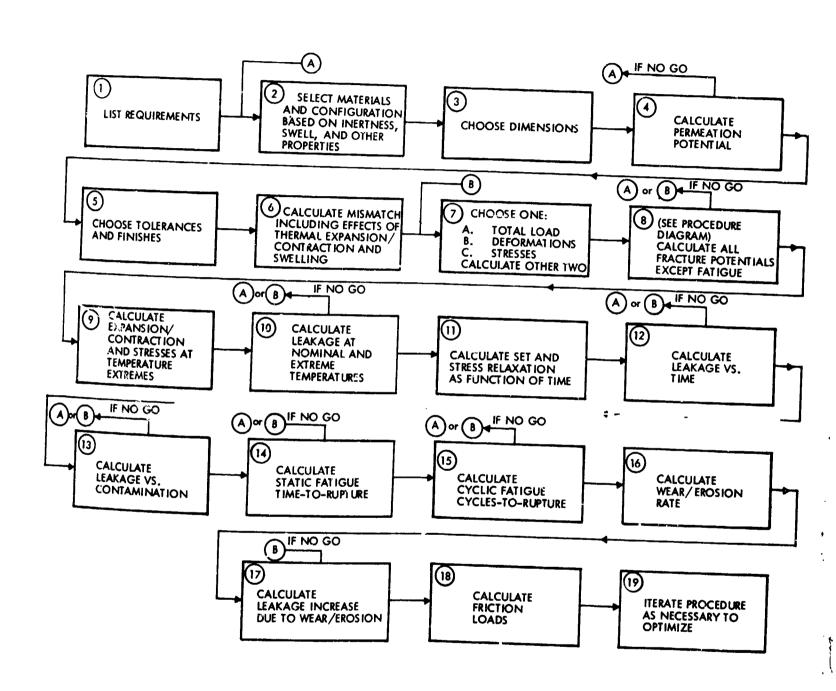
 $\sigma y = yield strength of polymer$

 S_i = shear strength of lubricating film

Go/No Go Criteria:

Return to Step 2 if allowable friction force criteria are not met.

STEP 19 - ITERATE AS NECESSARY TO OPTIMIZE
MAKE/DON'T MAKE CRITERIA:
Make if optimization is desired.
Inputs Required:
Criteria of acceptability of design
Procedure for calculating new values for new iteration
Available Methods:
Not applicable
Go/No Go Criteria:
Return to Step ② if criteria not met.



Southern Bridge Control March 1985

Figure 6.14 Seal Design Procedure
Diagram

COLDOUT FRAME

7.0 CONCLUSIONS AND RECOMMENDATIONS

This program has evaluated potential seal materials and characterized the seal materials suitable for use in propulsion system sealing applications. It is concluded that these materials can be applied to Space Shuttle Earth Storable Propellant seal applications with high confidence of success.

Two of the materials AF-E-124D and AF-E-411 are presently being used by TRW Systems in earth storable propellant flight systems and the other, AF-E-411A, is being used in a flight biological instrument package system as a valve seal.

Valve manufacturers are presently conducting some experimental evaluations of these materials for Space Shuttle applications.

7.1 Conclusions

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Although a number of seal materials have been used in the past for propellant service, all have exhibited problem areas which make them undesirable for extended service in Space Shuttle applications. The baseline material, and the material which is best characterized, Teflon, has shown many advantages due to its inertness in propellants.

For oxidizer service however, the elastomer AF-E-124D is considered to be generally superior, when used within specified conditions. The basic advantage are:

- Being an elastomer it is more tolerant to contamination and thereby exhibits longer life capability.
- Retention of sealing capability over a long term by increased compression set resistance. AF-E-124D retains resiliency after long periods of compression.

No other non-metallic material except Teflon, can be considered for N_2O_4 oxidizer seal service. Oxidizer incompatibility rules out other non-metallic materials. (Carboxynitroso Rubber is not yet sufficiently developed to be considered for use.) AF-E-124D has

demonstrated good oxidizer resistance. Constraints on the use of this material are primarily in the area of high temperature exposure. As operating temperature is increased, the mechanical properties are reduced. Above 200°F, consideration of reduced properties must be made. No permanent change occurs after return to room ambient temperatures.

For fuel service, both AF-E-124D and AF-E-411 are excellent materials and are recommended for Space Shuttle use. The optimum material is considered to be AF-E-411 for this service, since it exhibits less compression set than AF-E-124D. Since this material is currently being used for a fuel seal in flight applications, it would be the first choice where differing seal materials are permissible for fuel and oxidizer.

The recommended flushing and cleaning solution for AF-E-124D and AF-E-411 is isopropyl alcohol. Freon TF and trichlorethylene are not recommended since significant weight and dimensional changes occur as a result of exposure. The effects of very short term exposure to Freon TF have not been evaluated, and if use of Freon TF is necessary, evaluation of the planned exposure period effect on the elastomers should be determined.

As in all seal applications, the specific design must be oriented toward use of an elastomer, and to take advantage of elastomeric properties. When used in this manner these materials should be superior in performance to other seal materials for propellant service.

7.2 Recommendations

The seal materials recommended as a result of this program, have been characterized and evaluated to a large degree. There are factors which could not be determined in a relatively short term program. It is in this area that additional effort is recommended.

A primary concern in a seal material designed for long term service is the long term performance. A program of obtaining long term real time data is recommended to obtain data prior to the time components in operation will have reached that age.

There are a number of ways of accomplishing this. The most expeditious method of accomplishing this would be a test program approximately as follows:

Obtain property data on both stressed (compressive and tensile) and unstressed material samples, immersed in propellant as a function of time at room temperature. The tests would consist of compression set, tensile hanging weight tests, and mechanical properties as described below.

Compression Set Tests. O-ring compression set samples of fixed diameters should be placed in propellant storage at ambient temperature. Samples would be removed from propellant (e.g. 3 samples each of two sizes) each three month period for five or more years. Measurements of compression set would then be taken as soon as removed and then after an outgassing period.

Tensile Hanging Weight Tests. Triplicate or more specimens would be subjected to "hanging weight" tests in propellant at ambient temperature for five years or a selected period. The elongation as a function of exposure period to propellants would be determined at six month intervals. Mechanical properties would also be determined at these intervals.

<u>Mechanical Properties</u>. Triplicate unstressed specimens would be immersed in propellants with samples removed and mechanical properties measured every three months for five or more years.

This test series would provide long term compression set and mechanical properties data and is a minimum cost type of test which can be readily initiated since no special equipment (other than propellant storage) is required.

To provide additional data, all or part of the above test series could be placed in a thermally controlled environment and subjected to thermal cycles typical of anticipated Space Shuttle duty cycles.

Other recommended additional data which would provide added performance and long term prediction capability are:

- o Mechanical properties data while immersed in propellants as a function of propellant immersion period.
- o Conduct an analytical program to provide an extrapolation with relatively few data points from short term tests. Add and revise this extrapolation as data is required.
- o Determine valve and static seal configurations and place test fixtures, or actual hardware where available, in extended propellant storage conducting period leakage and functional tests.
- o Conduct additional system cleaning/flushing fluid tests and technique evaluation to determine degree of effect when using typical cleaning cycles with commonly used fluids such as Freon which were not recommended as a result of long term test. These fluids may be necessary for operational systems if cleaning/flushing operations are routinely conducted, and some flushing will be required for decontamination prior to maintenance.

In addition to these recommendations for acquisition of long term data the following general recommendations are made:

- When elastomers are to be used as a replacement for plastic seals, the identical design should not be used, but rather be designed for elastomeric use.
- (2) When using AF-E-124D in oxidizer, whenever possible do not clean and vacuum dry but leave the material exposed to N₂O₄. Since there is some swell upon exposure to propellant, this maintains a constant seal geometry.
- (3) The optimum valve type for use with an elastomer is a poppet type seal. This is primarily because of the high resiliency of the elastomer, which creates a higher degree of wear when in a sliding application such as a ball valve seal. When used in a ball valve, a retracting seal/ball is recommended.

APPENDIX A

ACKNOWLEDGMENTS

Many organizations have provided information and data during the course of this program. This support is appreciated and acknowledgement of these organizations is provided below. It is intended that all assistance be acknowledged and any omissions made are inadvertent.

Organization

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Air Force Materials Laboratory
McDonnell-Douglas Corp.
Martin-Marietta Corp.
The Marquardt Corp.
Aerojet Liquid Rocket Co.
Parker-Hannifin Corp.
Bell Aerospace Corp.
Space Division,
Rocketdyne Division

Rocketdyne Division Rockwell International Corp. Moog Inc. Fairchild Industries Inc. Futurecraft Corp. Wright Components Eckel Valve Co. Valcor Inc. Grumman Aircraft Corp. Sterer Engineering & Manufacturing Co. Accessory Products Co. Calmec Division of Ametek Inc. James Pond & Clark Inc. Division of Circle Seal Corp. Whittaker Corp. Dynasciences Controls Division Hydraulic Research & Manufacturing Co. E. I. du Pont de Nemours & Co. 3 M Company Pennsalt Corp. ICI America Inc. Dixon Corp.

Location

Houston, Texas
Huntsville, Ala.
Pasadena, Calif.
Wright-Patterson AFB, Ohio
Huntington Beach, Calif.
Denver, Colo.
Van Nuys, Calif.
Sacramento, Calif.
Irvine, Calif.
Buffalo, N. Y.
Downey, Calif.

Canoga Park, Calif.

East Aurora, N. Y.
Manhattan Beach, Calif.
City of Industry, Calif.
Clifton Springs, N. Y.
San Fernando, Calif.
Kenilworth, N. J.
Bethpage, N. Y.
Burbark, Calif.
Pacoima, Calif.
Los Angeles, Calif.
Anaheim, Calif.

No. Hollywood, Calif.

Valencia, Calif.
Wilmington, Del.
St. Paul, Minn.
Philadelphia, Penn.
Stanford, Conn.
Bristol, R.I.

APPENDIX B

EXCERPTS FROM ANALYSIS REPORT

The following Sections from the Analysis Report 72.4781.6-262, dated 6 November 1972 are included in this Appendix. Although the complete report is not included in this Appendix, much of the material contained in the remainder of Report 72.4781.6-262 has been incorporated and expanded in the body of the final report.

- 1.0 Introduction
- 2.0 Summary
- 3.0 Sealing Technology Survey

Report No. 72.4781.6-262 Contract No. 9-12729 DRL No. 2 DRD No. SE-423T Dale: 6 November 1972

ANALYSIS REPORT SPACE SHUTTLE SEAL MATERIAL & DESIGN DEVELOPMENT FOR EARTH STORABLE PROPELLANT SYSTEMS PROGRAM

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Prepared for NASA Manned Spacecraft Center Houston, Texas

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FOREWORD

The information contained in this report was obtained from a number of sources. Specific data was obtained through the cooperation of a number of contractors and government agencies. These sources are acknowledged in Appendix A of this report. Much information was obtained through informal discussion and it is intended that the organization be identified in the appendix. Any omissions are inadvertent.

With a survey of this type, particularly of state-of-the-art. information gathering is a continual process. Information is expected to be obtained after this report is issued. Data of significant value will be included in the future as a revision or appendix to this report.

Individual contributors at TRW include G. Howell, J. Martin as well as R. N. Porter.

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1. INTRODUCTION

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This report summarizes the results of the investigation conducted during Task I of this program. This Task, entitled Analysis, includes a survey of sealing technology to determine state-of-the-art in seal materials, seal designs, performance and seal material bonding techniques as related to earth storable propellants. The specific propellants of interest are, nitrogen tetroxide (N_2O_4), monomethyl hydrazine (MMH), hydrazine (N_2H_4), unsymmetrical dimethyl hydrazine (UDMH), and a 50/50 mixture of UDMH and N_2H_4 . Seal applications are primarily closure, static and dynamic.

In the survey effort, past and current propulsion systems are one category investigated, and advanced or development systems or techniques another.

Also included in the Analysis Task is the determination of critical material and design parameters related to seal design and performance. Evaluation of all known factors influencing seal behavior are included in this investigation, and a determination of the relative criticality.

The summation of these efforts results in a determination of present seal state-of-the-art capabilities and limitations, which is the last phase of the analysis task.

The results of these activities are reported in Section 3 State-of-the Art Survey; Section 4 Critical Material and Design Parameters, and Section 5, State-of-the Art Capabilities.

Section 2 provides a brief summary of the results of each effort and conclusion. References are listed in the appendices.

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2. SUMMARY

Application data from the testing of successful flight systems has shown that present state-of-the-art sealing technology is, in general, adequately satisfying todays earth storable propulsion system requirements. Typical of these requirements are soft seat or poppet valves using Teflon as used for Apollo Service Module and Lunar Module RCS engines and Teflon ball valve seals used in the Lunar Module Ascent and Descent engines. Typical monopropeliant applications are metal to metal sealing in Intelsat IV; Teflon sealing in Transtage RCS and P-95 RCS; and ethylene-propylene rubber elastomers in Intelsat III, and numerous military satellites.

In many cases, the cycle and operating life requirements are rather low, but there are cases where high operating and cycle life is necessary. Satellite applications of small valves is a primary example where a five year life has been demonstrated. However, the development and manufacturing costs of many of these valves has been excessively high. The forthcoming Space Shuttle propulsion systems add a new dimension to aerospace valve applications, including multiple reuse, refurbishment, and production techniques more consistent with commercial aircraft philosophy. These new criteria warrant, if not dictate, the development of new seal material and design philosophies.

A review of present and advanced propulsion system capability indicate that seals in monopropellant systems are more capable of meeting long term, high cycle life requirements typical of Space Shuttle applications than are current bipropellant system seals. The primary reason for this is oxidizer compatibility problems, since very few materials are compatible with N_2O_4 . A basic goal has been to use the same seal material on both sides of bipropellant valves, which limits the fuel side to materials compatible with N_2O_4 . The basic material used, therefore, has been Teflon, with some other materials being used in limited applications. Teflon has been and is an excellent seal material, since it does not react with N_2O_4 . However, Teflon does have limited cycle life capability due to "cold flow" or permanent set, which eventually allows leakage even when fully contained. This is more evident in closure seals than in static applications.

The high cycle life, long term seal problem is most critical with N_2O_4 , although hydrazine base propellants are not without long term problems. Development of a compatible seal material for $N_2^{0_4}$ service, having good restoration properties typical of an elastomer is a major step required for earth storable bipropellant Space Shuttle applications. New design techniques can undoubtedly increase the capability of Teflon seals, but the degree of improvement would not appear to satisfy long term, low maintenance needs. The most promising improved seal materials presently in early or advanced development as indicated by this summary are those in work at TRW Systems. No other significant advanced materials were identified through discussion and contact with many sources. Since new materials do represent a potential technical sales advantage it is understandable that many companies may be reluctant to identify them. However, through contacts within the chemical community as well as in seal design development areas, it appears to be generally accepted that the TRW development materials are currently the most advanced. These materials are AF-E-124D, a fluoroelastomer compatible with $N_2 O_4$ and hydrazine base fuels, and AF-E-102 and AF-E-411 ethylene-propylene terpolymers with "HYSTL" additives for hydrazine base fuel service.

To say that without these new materials, the Space Shuttle requirements cannot be met, would be a mis-statement and an injustice to the excellent seal designers both in government and industry. However the cost, and ease with which these requirements are met are a function of whether new materials can more readily be applied. Contamination sensitivity will remain a major problem with materials other than elastomers, and greatly influences system capability, particularly in closure and dynamic seals. For this reason, advanced elastomer development is highly desirable.

Cycle life limitations are more evident and potentially severe with large valve seal designs than with smaller types. These cases emphasize the mechanical limitations of existing seals and the need for improved materials. Cycle life of 25,000 cycles or less is typical of existing large valve seals, while requirements of 100,000 cycles or greater are required for Space Shuttle applications.

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Existing static seal data do not provide confidence for long term application, primarily because of the scarcity of applicable data. Most static seals are metallic pressure energized seals, in most cases with a coating of soft metal or polymer. These are suitable only for limited service. O-rings, the most common seal type, perform to differing degrees dependent on material characteristics, but all will take a permanent set over extended periods of time. This will, to some degree, restrict system life capability. Most existing systems requiring long service life use permanent welded or brazed joints for maximum reliability. Improved elastomers and designs are required for separable connectors for use on long term propulsion systems.

The material and design parameters which are critical to storable propellant seals were found to be generally similar to those identified for oxygen-hydrogen systems in TRW Report 72.4781.6-154. Although the propellants are different and the temperature range less severe the basic sealing mechanisms are identical. The degree of criticality is not altered, although the effects of compatibility are generally more pronounced. The parameters considered of major importance are shown in Tables 2-1, 2-2, 2-3 and 2-4 for poppet type, ball valve, static and dynamic seals. The degree of criticality is indicated for given conditions, but is as dependent on seal design as other factors. While an absolute list ranking seal parameter criticality for all conditions cannot be prepared, certain parameters rank high in all cases. Inertness (propellant compatibility) and permanent set are primary examples.

The basic conclusions reached during this investigation are that materials not presently in current state-of-the-art service are highly desirable, if not absolutely necessary to meet the long term goals for Space Shuttle propulsion systems. These materials require design characterization for proven seal applications and improved analytical techniques for optimum seal design as related to the most critical parameters for the Space Shuttle seal requirements. These needs are essential for proper design, development and production of seals for storable propellant propulsion systems.

The results of this phase of the program indicate that the continuing program evaluation should emphasize the following basic areas:

- Develop and/or characterize new elastomeric seal materials compatible with $N_2 O_4$ and hydrazine base propellants.
- Improve analytical methods of seal design and performance evaluation.
- Provide improved seal concepts using improved materials and conduct confidence and characterization tests.
- Continue investigation of advanced seal materials from industry sources for possible inclusion in the program.

Table 2-1.

		Leakage	Control				Durability	ity	
		Seal Mai	terial		Design	Seal Materia		Design	5
		Effect	t on Seal Performance if		Parameter is:	Effect	on Seal Perfor	Effect on Seal Performance if Parameter is:	meter is:
Parameter	Desired Property	Morst Case	Deficient But Not Morst Case	Not Corsidered	Minimum Consideration	Worst Case	Deficient But Not Mors: Case	Not Considered	Minimum Consideration
Inertness (Propellant and Space Environment Compatibility	High	Critical	Critical	Critical	Critical	Critical	Critical	Oritical	Critical
Contamination Sensitivity	Low	Major	Major	Cri tical	Major	Critical	Major	(ritical	Critical
Permanent Set	LOW	Major	Minor	Critical	Major	Critical	Major	Gritical	Critical
Sealing Load	LOW	Minor	Minor	Major	Minor	Major	Major	Oritical	Major
Material Temperature Effects	LO#	Major	Minor	Major	Major	Major	Major	(ritica)	Major S
AP Sensitivity (Mech.) Properties, etc.)	Low	Major	Minor	Major	Minor	Major	Major	Oritical	Major
Permeation	Low	Minor	Minor	Minor	Minor	Minor	Minor	Tinor	Minor
Surface Finish (Seal)	High	Minor	Minor	Major	Minor	Minor	Minor	kajor	Minor
Surface Finish (Gland or Seat)	High	N/A	N/A	Major	Major	 Major	Minor	(Pritical	Major
Kardness	High	Minor	Minor	Major	Minor	Major	Minor	ajor	Minor
Mismatch	LOW	Minor	Minor	Minor	Minor	Major	Minor	ajor	Minor

Table 2-2.

		Lea	Leakage Control				ð	Durability	
		Sez	Seal Material		Design	Š	Seal Material		Destan
		Effect	on Seal	Performance if	arameter is:	Effec	Effect on Seal Performance	=	Parameter is:
Parometer	Desired Property	Hors t Case	Deficient But Not Worst Case	Not Considered	Minimum Consideration	Morst Case	Deficient But Not Worst Case	Minumum Consideration	Not Considered
Inerthess (Propellant and Space Environment Compatibility)	High	Critical	Critical	Critical	Critical	Critical	Critical	Critical	Critical
Contamination Sensitivity	Low	Critical	Major	Critical	Critical	Critical	Critical	Critical	Critical
Permanent Set	Low	Major	Major	Major	Major	Hajor	Major	Critical	Critical
Sealing Load	Low	Major	Major	Major	Major	Major	Major	Critical	Critical
Material Temperature Effects	LOW	Major	Major	Critical	Major	Major	Major	Critical	Critical
AP Sensitivity (Mechanical Properties, etc.)	76	Critical	Critical	Critical	Major	Major	Major	Critical	Critical
Permeation	Low	Minor	Minor	Minor	Minor	Minor	Minor	Minor	Minor
Surface Finish (Seal)	Hfgh	Critical	Major	Critical	Major	Major	Major	Major	Critical
Surface Finish (Gland or Seat)	High	N/A	N/A	Critical	Major	Critical	Critical	Critical	Critical
Hardness	High	Major	Major	Major	Major	Major	Major	Major	Major
Stroke (Silding)	Low	M/A	N/A	Critical i	Major	Major	Major	Critical	Critical

Table 2-3.

Summary of the Most Critical Parameters for Non-Metallic Static Seals

		Leakag	Leakage Control (a	(and Durability)	
- A W.		Seal Materia	teriai	Design	ub
		Effect	on Seal Perf	Effect on Seal Performance if Parameter is:	ter is:
Parameter	Desired Property	Worst	Minimum But Not Worst Case	Not Considered	Minimum Consideration
Inertness (Propellant and Space Environment Compatibility)	High	Critícal	Critical	Critical	Critical
Contamination Sensitivity	FOM.	Critical	Critical	Critical	Critical
Permanent Set	LOW	Major	Major	Critical	Major
Material Temperature Effects	FOM.	Major	Major	Critical	Marion
Perseation	LOW	Critical	Major	Critical	Major
Surface Finish (seal)	High	Major	Major	Major	Major
Surface Finish (Gland or Seat)	Higi	N/A	N/A	Critical	Major
Hardness	High	Major	Minor	Major	Major

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Table 2-4.

	-		Summary		of Critical Parameters for Non-Metallic Dynamic Seals	-Metallic Dy	mamic Seal	w		
			Le	eakage Control				Durability	ţ	
			Seal Mater	lal	Design		Seal Material	terial	De: 1gn	
			Effe	Effect on Seal Performance if Parameter is:	formance if Para	umeter is:	Ef	Effect on Seal	Performance 1	Performance if Parameter is:
	Parameter	Desired Property	Worst Case	Deficient But Not Worst Case	Minimum Consideration	Not Considered	Worst Case	Deficient But Not Norst Case	Not Considered	Minimum Consideration
	Incremess (Propellant and Space Environment Compatibility)	Hfgh .	Critical	Critical	Critical	Critical	Critical	Critical	Critical	Critical
	Contamination Sensitivity	Low	Critical	Oitical	Major	Critical	Critical	Major	Critical	Major
B-1	Permanent Set	LOW	Major	Minor	Major	Critical	Critical	Major	Critical	Major
19	Sealing Load	LOW	Major	Major	Major	Critical	Critical	Major	Critical	Major
	Material Temperature Effects		Critical	Major	Major	Critical	Critical	Major	Critical	Major
	Permeation	Low	Minor	Minor	Minor	Minor	Minor	Minor	Minor	Minor
	Surface Finish (Seal)	High	Major	Major	Critical	Critical	Critical	Major	Oritical	Major
	Surface Finish (Gland or Seat)	High	N/A	N/A	Critical	Critical	Critical	Major	Critical	¥a∮.
	Hardness	High	Major	Mfnor	Major	Major	Major	Major	Major	Major
	Stroke (Sliding)	Low	N/A	N/A	Critical	Critical	N/A	N/A	Critical	Oritical
						_				

SEALING TECHNOLOGY SURVEY

A sealing technology investigation was conducted as part of the analysis task to determine and document state-of-the art. This investigation encompassed past and existing propulsion systems and also newer systems or development programs. The areas of interest include seal materials, seal designs, performance, and bonding technology. Although all known systems have been considered, not all are presented here either because of similarity, lack of data, or the proprietary nature of the data. As would be expected, (and experienced during the survey of oxygen-hydrogen seals for Contract NAS 9-12500) specifics of seal design are closely guarded by many companies since this is one of the critical areas determining valve or seal performance. As such, no data known to be proprietary or restricted by a source of information has been included.

Section 3.1 identifies typical past .nd existing propulsion system components, seal types and an evaluation of designs and capabilities. The bulk of the state-of-the art information comes from these systems since they are operational and have the greatest history. Both bipropellant and monopropellant systems are covered.

Section 3.2 discusses more advanced systems or techniques which are either in development currently or have been developed but not used operationally. This can be a very fine line, since one flight will classify an application as operational, while only a qualification program may not. These classifications have not been strictly adhered to since alternate sources or completely qualified components and systems should be approximately equivalent to flight systems.

Materials in current use or being developed are identified in Sections 3.1 and 3.2. These materials are discussed at greater length in Section 3.3 along with any which should be considered as a result of current development efforts.

Methods of obtaining data included using the WESRAC (Western Research Applications Center) literature search compiled for Contract NAS 9-12500 (included in report 72.4781.6-168), TRW reference library and vendor files, and primarily, contact with systems and/or component manufacturers, and users. Much of the detailed information needed has not been documented to

the extent that it can readily be transferred, and in these cases verbal data were used to establish design type, capability and performance.

3.1 PAST AND EXISTING PROPULSION SYSTEMS

A survey of all known propulsion systems was made. A summary of important sealing components in selected systems is shown in Tables 3-1 and 3-2. Table 3-1 shows typical bipropellant system valve types and requirements or capability. The propulsion systems addressed are Gemini, Apollo LM/AE DE & SM, Transtage, Lunar Orbiter, Minuteman III Post Boost Propulsion System, Delta, Agena, Saturn SIVB, Titan III, MMBPS, and ISPS. These are not all the past and existing storable propellant systems, nor are all the valves in each system covered in detail. However, the valves listed are considered representative of state-of-the art in those systems with varying valve sizes included and the systems tabulated summarizing past and existing state-of-the art. Specific valve seal designs are briefly described identifying characteristics of design where possible. Specific details of manufacture such as surface finish, seal material conditioning and seal loads are not provided in most cases since these are considered proprietary by many manufacturers.

This table and attendant state-of-the art in bipropellant systems indicates that Teflon is predominantly the state-of-the art valve seal material for most applications. Other non-metallic materials have been used such as Kel-F, polypropylene and Kynar, but in a limited number of cases. The problems of material compatibility with N_2O_4 severely limits the choice of material. Metal-to-metal seals have also been used, but also for a limited number of applications.

Table 3.2 provides a summary of monopropellant hydrazine valves and capabilities. Although the wide use of monopropellant hydrazine for propulsion systems is relatively new there are a large number of systems that have been developed primarily for RCS or attitude control of satellites. The primary valve applications are for thruster valves, and propellant tank isolation. Most of the applications are for low (less than 10 $1b_{\rm f}$) thrust, although applications to 300 $1b_{\rm f}$ have been accomplished.

1					ļ	j	See	ı _	Sei	ıt			Rate ec.	
Vehicle or Stage & Subsystem	Engine & Supplier	Fluid	Valve Type	Valve Menufacturer	Pressure psig	Flow Rate 1b/sec	Material	Config.	Material	Config.	Test Fluid	Test Press psig	Allow. Rate SCOH	Cycle Life Spec.
Mnuteman III PBPS (Axial)	LR-123-NA-1 (Rocketdyne)	N ₂ O ₄	Torquemotor Poppet	Moog	247	0.67A 0.424	Teflon TFE Teflon TFE	Flat	17-7PH 17-7PH	Flat Flat	GM2	247	5.0 5.0	10,000
Minuteman III PRPS (RCS)	LR-125-BA-1 (BAC)	N2O4 HHH	Torquemotor Poppet	Moog	247	.069 .055	Teflon TFE Teflon TFE	Flat Flat	17-7PH 17-7PH	F]+t Flat	G#2	247	5.0 5.0	10,000
tariner Mars 71	RS-2101 (Rocketdyne)	N204 MMH	Torquemotor Poppet	Moog	300	0.68	Teflon TFE Teflon TFE	Flat Flat	17-79H 17-79H	Flat Flat	GH2	300 300	5.0 5.0	
lpollo SM RCS	R-4D (Marquardt)	N204 A-50	Solenoid Poppet	Marquardt	185	0.24	Teflon TFE	Beveled Disc	Stellite No. 6	Conical Poppet	GH2	200	5.0	
pollo LM CCS	R-4D-2 (Marquardt)	N204 A-50	Solenoid Poppet	Marquardt	195	0.24	Teflon TFE	Beveled Disc	Stellite No. 6	Conical Poppet	GN2	200	5.0	
lpollo system isolation	N/A	N204 A-50	Latch Solenoid Poppet	Parker	250	1.0	Teflon TFE	Flat	304L	Flat	GHe	250	10.0	1,000
Mpollo LA Ascent	(Rocketdyne)	N ₂ 04 A-50	Ball (Quad)	BAC	205	7.0 4.3	Teflon TFE	Ring	302	Ba17				
Apollo LM Descent	(TRM)	N204 A-50	Ball (Quad)	Whittaker	231	19,9 12,5	Teflon TFE	Lipseal	17~4PH	Partial Ball	GH2	250	0.1 SC1M	
Apollo LM Prevalve	R/A	A-50	Soleroid Poppet	Valcor	280	12.5	EPR	Retained 0-Ring	304L	Conical	GH2	250	10.0	
Apollo SM	AJ10-137 (AGC)	N204 A-50	Sall (Qued)	AGC	2 25	38 24	Teflon Glass- filled	Lipseal	17-4PH	Ball	ON ₂	150	200 (500/ valve)	1000
unar Orbiter	R-4D (Marquardt)	N204 A-50	Poppet	Marquardt	190 190	0.24 0.12	Teflon TFE	Beveled Disc	Stellite No. 6	Conical Poppet	GAIZ	200	5.0	
OMBPS	URSA-100R (TRM)	N204	Torquemotor Poppet	Moog	300	0,18 0,11	Teflon TFE	Flat	17-7 9 H	Flat	CM2	235	5.0	25,00
Agéna	LR-81-BA (BAC)	IRPNA UDMH	Poppet Poppet	BAC BAC	1 000 900	36.0 15.0	Teflon EPR	Conical Conical	304L 304L	Conical Conical	GH2 GH2	1000 900	60 60	500 500
Agena - Propellant Isolation	N/A	IRFNA UDM	Gate	Whittaker	100		Teflon	Cartridge	CRES	Flat Belleville	CH2	100	6x10 ⁶ sccsec	5,000
Titan Transtage	AJ 10-138 (Aerojet)	N204 A-50	Poppet	AGC	250	27:5 17:3	Teflon	Lipseal	CRES Alum		Gr.	250	30	5,00
Titan Stage 2	LR91-A5 (Aerojet)	N204 A-50	bu tterfly	AGC AGC	1113 1195	1000 500	Kel-F Hylon	Lipseel Lipseel	17-4PH 17-4PH	Butterfly Butterfly	CHZ	1100	1900	100
Titan Stage I	LR87-A5 (AGC)	N204 A-50	Butterfly	AGC	1057 1358	440 220	Kel-F Nylon	Lipseal Lipseal	17-4PH 17-4PH	Butterfly Butterfly	CHZ	1100	1900	100
Titan Transtage RCS	SE-9 (Rocketdyne)	N204 A-50	Poppet	Rocketdyne	305									
Com ini	SE-6 (Rocketdyne)	N204 1994	Poppet	Rocketdyne	295	.017	Teflon FEP		Stellite No. 3			1		}
Genini OAMS	SE-7 (Rocketdyne)	N204 1991	Poppe t	Rocketdyne	295	8:1					1			
Apollo CH	SE-8 (Rocketdyne)	N204 MAH	Poppet	Rocketdyne	300	0.2 0.1	Teflon	Ring	CRES	Poppet	GM2	300	0.2	
Saturn IVB RCS	700800 (TRM)	N204 MMH	Solenoid Poppet	Valcor	195	0.31	Teflon	Flat	304L	FlatFoppet	Liq	275	0.021	10,0
Saturn IVB VCS	SE-7B (Rocketdyne)	N204 HMH												
i									 					
			{				{		}	{				

RCS - Reaction Control System OMRS - Orbital Attitude & Maneuvering SR - Service Module LH - Lunar Module CR - Command Module

CRES - Corrosion-resistant Steel SCCH - Standard Cubic Centimeters per Hour

PPS - Post-Boost Propulsion System NAPS - Rulti-Hission Bi-Propollant System ICC - Aerojet General Corporation IAC - Ball Aerosystems Corp

Rocketiyne - Rocketiyne Division, North American Rockesii Corp. Harquardt - Harquardt Corp. The - The System Hoog - Noog Incorporated Parker - Parker Hemsifin Corp.

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Table 3.1. Representative Bipropellant System Valves

FOLDOUT FRAME

					5
Vehicle or Stage	Engine or System Supplier	Valve Type/ Function	Pressure psig	Flow Rate 1b/sec	Materia
Intelsat III	TRW	Poppet- Thruster	600	0.015	EPR
Pioneer F&G	TRW	Poppet- Thruster	600		EPR
DSP	TRW	Poppet- Thruster	600	0.015	EPR
DSP	TRW	Poppet- Thruster	20		EPR
DSCS II	TRW	Poppet- Thruster	600	0.015	EPR
Intelsat IV	Hughes	Poppet Torque Motor Thruster	365		Tungsten Carbide
Intelsat IV	Hughes	Poppet Isolation	300		Teflon
ATS F&G	Rocket Research	Poppet- Thruster	400	0.00045	Teflon TFE
ETS A	Rocket Research	Poppet- Thruster	400	0.0022	Teflon TFE
P-95	Rocket Research	Poppet- Th <i>r</i> uster	300		Teflon TFE
P-95 (Alternate)	Rocket Research	Poppet- Thruster	300		Teflon TFE
Transtage	Rocket Research	Poppet- Thruster	400	0.117	Teflon TFE
RAE-B	Hamilton-Standard	Poppet- Thruster	225		Tungsten Carbide
SMS	Rocket Pesearch	Poppet- Thruster			
IDCSP/A	Hamilton-Standard	Poppet- Thruster			
Skynet (NATO)					
NRL Explorer	Hamilton-Standard				}
Canadian ATS	Hamilton-Standard		1		
NRL HPM	Rocket Research		{		
827	Rocket Research				1

COLDOUT FRANCE

İ		Seal		Seat	:	Leal	k Rate Spec	if.		
re	Flow Rate 1b/sec	Material	Config	Material	Config	Test Fluid	Test Pressure psig	Allow. Rate SCCH	Cycle Life Spec.	Valve Manufacturer
	0.015	EPR	Flat	304L	Flat	GN2	600	0.5	60,000	Fairchild Industries
		EPR	Flat	304L	Flat	GN2	600	0.5		Allen Design Inc.
	0.015	EPR	Flat	304L	Flat	GN2	600	0.5	60,000	Fairchild, Allen Design, Parker-Hanni
		EPR	Flat	304L	Flat	GN2	20	0.5	1 x 10 ⁶	Allen Design Inc Gas (Decomp N ₂ H ₄)
	0.015	EPR	Flat	304L	Flat	GN2	600	0.5	60,000	Fairchild Industries, Parker-Hannifin
		Tungsten Carbide	Flat	Tungsten Carbide	Flat	GN2		5.0		Hydraulic Research Corp.
		Teflon	Ring	300 CRES	Conical	GN2	300	2.0	1,000	Carleton Controls
	0.00045	Teflon TFE	Flat	304L	Flat	GN2	400	1.8	1 x 10 ⁶	Parker-Hannifin
	0.0022	Teflon TFE	Flat	304L	Flat	GN2	400	5.0		Parker-Hannifin
		Teflon TFE	Flat	304L	Flat	GN2	300	1.0	1 x 10 ⁵	Parker-Hannifin
	 	Teflon TFE	Flat	304L	Flat	GN2	300	1.0	1 x 10 ⁵	Moog Inc.
	0.117	Teflon TFE	Flat	17-7PH	Flat	GN2	360	5.0	72,000	Moog Inc.
		Tungsten Carbide	Flat	Tungsten Carbide	Flat	GHe	210	5.0		Hydraulic Research Corp.
				Carpide				ļ		Parker-Hannifin
										Hydraulic Rese. th Corp.
										Hydraulic Research Corp.
÷										Wright Components Wright Components
										Parker-Hannifin
	! 4		1						}	Parker-Hannifin

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Table 3.2. Representative Monopropellant Hydrazine System Valves

8-25 FOLDOUT FRAM

2

The type of valve used is a poppet-type either coaxial solenoid or torquemotor with single or redundant sealing elements. Materials in general use for valve seals are: Teflon, ethylene-propylene rubber, (EPT) ethylene-propylene terpolymer-HYSTL, (EPT/HYSTL) and various metal combinations. Butyl rubber could also be used and probably has but no specific application was identified. More complete materials discussions are provided in Section 3.3, materials.

3.1 | Bipropellant System Sealing

A number of successful operational bipropellant systems are in existence, some of which are shown in Table 3-1. All of these systems are typified by relatively short life cycle requirements, at least as indicated by specification values. Since, in most cases, no requirement exists for a higher cycle life, the maximum cycle life is unknown, but it is probably valid to assume the same order of magnitude.

Some of the valve seal applications are described and discussed below as an indication of the types of seal designs presently in use in typical state-of-the art systems.

Apollo Service and Lunar Module RCS Engine Valves

This valve, referred to as the Model R-4D valve, was developed by the Marquardt Company and was qualified and flown as part of their Apollo Service and Lunar Module RCS engines. In addition it has been qualified and flown on the Lunar Orbiter and classified satellites. Currently it is under test at JPL for use in the Mariner '73. In these applications the propellants were (are) N_2O_4 , Aerozine-50, and monomethyl hydrazine. The total amount of development expended on these valves has been very large and they are very expensive but the results, as summarized below, are outstanding.

Figure 3-1 shows the seat/seal details of the valve. By beveling the Teflon disc at 43 degrees and the metal poppet at 45 degrees a progressive contact is achieved which starts at the "upper" edge and ends at the "lower" edge where metal-to-metal contact occurs between the poppet and the beveled metal support beneath the Teflon disc.

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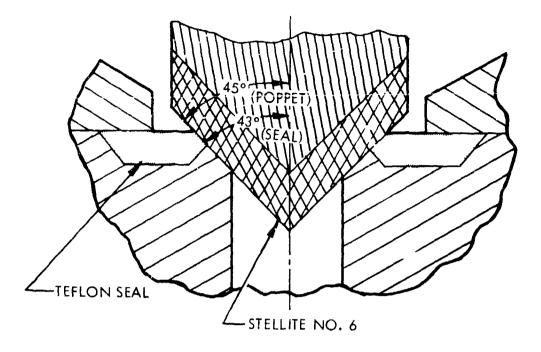


Figure 3-1. Marquardt R-4D valve seat detail

In the fully-closed position the spring applies a 2.75-pound load; the poppet is pressure unbalanced so fluid pressure will increase loading by about 0.038 times the pressure. This force generates an apparent sealing stress of approximately

$$\sigma_{s} \approx 72.5 + P$$

both the seal and poppet surfaces are finished to approximately 8μ in.

Leakage rates specified for most applications was 5 scc per hour of gaseous nitrogen at pressures up to 200 psi. Measured leakage rates have been less than 0.3 scc/hr in the vast majority of valves tested. Specified service temperature range is +32 to +200°F.

Qualification for the Apollo application entailed a great amount of testing, the results of which are readily available to NASA in Reference 61, and so will not be repeated here.

The most recent testing has been carried out at JPL as part of the Mariner '73 project. For that application the allowable leak rate is 3 scc/hr of $\rm GN_2$ at 380 psig. Three valves used in 65 firings for a total firing time of 7500 seconds yielded a maximum leakage rate of 0.68 x 10^{-4} scc/sec of helium gas at 300 psig. One valve was put through a random vibration test at 5.5 g's rms for one minute along each of three axes (including the valve poppet axis) after which the leak rate was 0.75 x 10^{-4} (same units) at both 10 and 300 psig. After 1000 dry cycles with a vacuum downstream a valve was leaking at the rate of 0.03 x 10^{-4} (same units) at 300 psig. Results of a six-month soak in hydrazine with vacuum downstream were not available at this writing but apparently the accumulated leakage was about 1/100th of the acceptable rate.

Two valves were subjected to the subsystem Type Approval testing. Vibration testing included one minute along each axis of 10 g's rms random, sine wave sweeps along each axis at one octave per minute at 0.75 g's rms for 5 to 35 Hz, 6 g's rms from 35 to 300 Hz, and 12 g's rms from 300 to 2000 Hz, plus a combined frequency test increasing at 24 db per octave from 10 to 50 Hz to a level of 0.28 g 2 /Hz, a constant 0.28 g 2 /Hz at 750 Hz and above that at 12 db per octave to 2000 Hz. No increase in leakage rate above the initially measured amount of approximately 1 x 10 $^{-6}$ was detected. These two valves were also put through the Flight Acceptance testing with the same results.

Five valves fabricated in 1969 were subjected to the Flight Acceptance testing. This test series included vibration along three axes at these levels: 6 g's rms random for 20 seconds, sine wave sweeps at 3 octaves per minute at 0.5, 4, and 8 g's rms for the same frequency bands cited above for TA testing, and combined frequency tests as outlined above but at 0.1 $\rm g^2/rms$ and 0.022 $\rm g^2/rms$ rather than at 0.28 and 0.063 $\rm g^2/rms$. Following these tests the leak rates were in the range of $\rm 10^{-7}$ scc/sec of helium at 435 psig.

From these results at JPL it may be inferred that the R-4D valve seat is very leak-tight under the conditions of test.

Minuteman III Post Boost Propulsion System (PBPS)

This propulsion system is somewhat unique in that as a strategic weapons system, it must be capable of long term storage over a range of temperatures and conditions, and yet be able to perform on command with no pre-launch checkout similar to most spacecraft launch or propulsion systems. This system is the "fourth stage" of the Minuteman III system.

Most of the system is welded or brazed with metallic propellant isolation devices during storage. Therefore, although the propulsion system may be stored for five years prior to use, the propellant is isolated from non-metallic seals during this period. In this system, to insure that no propellant degradation occurs during storage this valve (and the RCS valves) see no propellant prior to flight. All flow data is obtained with reference fluids.

Two types of valves are used in this system for propellant control for the axial engine and to the RCS engines. The axial engine valve is a torquemotor valve utilizing a soft Teflon poppet sealing on a thin metal seat land and the RCS engines are torquemotor operated with a Teflon seal in the seat. Although the specific details of design and construction are considered proprietary to the valve manufacturer, the sealing technique is as indicated schematically in Figure 3-2. In this design the poppet incorporates a Teflon ring, which is mechanically retained. The metal seat land is sized to provide adequate seal loading, but combined with the entrapped Teflon configuration, is not high enough to result in excessive cold flow as evidenced by successfull operational history. The metal seat finish is important while the surface finish of the poppet is of lesser importance. Of major importance would appear to be manufacturing techniques for the Teflon and seat including parallelism of the mating surfaces. The design cycle life of 10,000 cycles and leakage specification of 5.0 scc/hour would appear conservative for this seal design.

The RCS valve seal is shown schematically in Figure 3-2. A Teflon seal is incorporated in the stationary seat element in this design. Mechanical seal retention is generally as shown in the figure with two concical surfaces retaining the Teflon, with sufficient protrusion above

the surface effect a seal against the flat poppet face. A large data base has shown this technique to be reliable for the 5.0 scc/hr specification leakage rate and required cycle life of 10,000 cycles. This design is also highly dependent on processing and fabricating techniques for the metallic and non-metallic components.

The two sealing techniques indicated by Figures 3-2 and 3-3 cover the seal types for a number of systems. In particular, Figure 3-2 typifies the sealing concept used for Mariner Mars '71, MMBPS, and ISPS, bipropellant systems; and Transtage and P-95 alternate valves.

The Minutemen PSFS valve seal design has been qualified to the following conditions and subjected to flight tests:

	Axial Engine Valve	RCS Engine Valve
" one tent	N ₀ 0 _a per ST1001-047006 (BAC)	NoOg per \$71001=047006 (3AC)
······································	11.7 Box Wileb South.	mn nen MIX-9-27404
<u>va Life</u>		
Storage	5 years minimum	5 voans minimum
Ovoles, pressurfized	10,000 (1000 drv)	1: ,300 (1000 dry)
to any of ing		
a et mad	+20 to +150°F	+20 tc +150°F
- `. , •	+60 to +80 f	+rg th kinghe
	(except +100°5 heat soak pack)	(except +100 f soak back)
r. ossure		
lperating	247 psig	247 psig
£ + 20 +	435 psi3	435 psig
200 2	585 psig	Shō nsiq
En roments		
<pre>.ibration (Operating)</pre>	2	3.0g ² /Hz narrow band, or 0.6g ² /Hz broad band (29.85 ms)
Pan to	$0.3g^2/Hz$ (max) to 2000 cps	
S. sucoidal	1.75g nms (max)	1/A
Srock	45g peak (max)	45g peak (max)
Ermidity	0 - 95% R.H.	υ = 95% R.H.

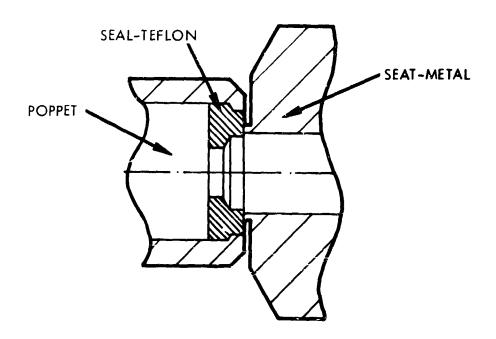


Figure 3-2. Schematic Poppet-Seat Configuration MM III Axial Engine

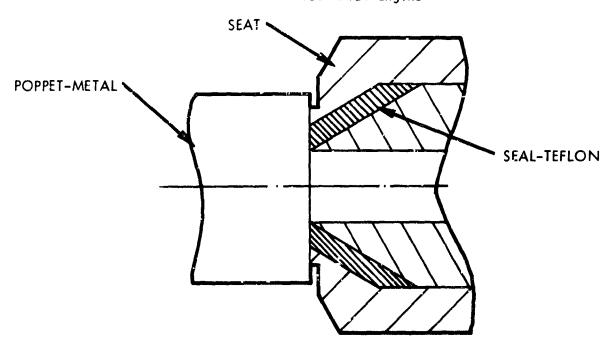


Figure 3-3. Schematic Poppet-Seat Configuration MM III RCS Engine

Apollo Latching Valve

This valve was used on the fuel and oxidizer propellant storage systems of the LM. The basic purpose was to provide shutoff capability for propellants. Approximately 800 units have been made and the valve has seen extensive flight service on Apollo. Figure 3-4 shows the poppet sealing mechanism, although not necessarily to scale. In this valve the poppet is of a pressure balanced design allowing close control of the seal load, primarily through the spring force. The Teflon is mechanically retained in the stationary seat area of the valve. Teflon seal stresses are controlled by means of a metal stop which limits the Teflon deformation and seal stresses. By controlling the free height of the Teflon above this stop, excessive deformation under load (cold flow) is eliminated, although normal compression set occurs as with any polymeric material.

The qualification requirements for this valve are shown below:

<u>Propellants</u> - N₂O₄, Hydrazine, UDMH

Design Life - 2000 cycles (min)

Temperature - -65°F to +240°F

Pressure

Operating - 250 psig
Proof - 750 psig
Burst - 1050 psig

Environments

Vibration

Random - $0.6g^2/cps$ (max) Sine - 10g peak (max)

Shock - 15g peak (max)

Humidity - 100%

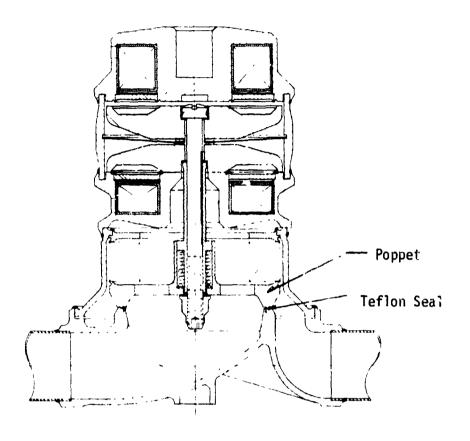


Figure 3-4. Apollo Latching Valve Showing Seal Configuration

Lunar ibdule Descent Engine Valve

A quad-redundant ball valve was designed and manufactured by Whittaker Corp. for use in the TRW. Apollo. Lunar Module Descent Engine (LMDE). This shutoff valve was designed for N_2O_4 and A-50 service over a fluid temperature range of +40 to +120°F. Nominal valve flow passage diameter was 1.125 inches.

The rotors (partial spheres of 1.93-inch diameter) were made of 17-4PH CRES and polished to a 4μ -inch finish. Dual lipseals of Grade 7A Teflon in a "U" shaped configuration acted as valve and static seals (see Figure 3-5). Spherical lands of about 40 to 60 mils width were pressed against the rotors with a force of 30 to 38 pounds by Belleville springs, generating an apparent sealing stress of 88 to 170 psi at zero fluid pressure. These lands were specified to have a 32μ -inch finish. Specification leakage rates were 10 scc/hr of GN_2 at 235 psig.

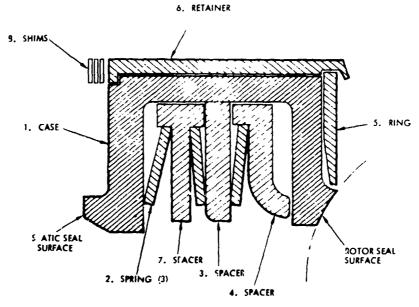


Figure 3-5. LMDE Shutoff Valve Seal Cross Section

Relaxation and wear was found to reduce the load to between 28 and 30 pounds, yielding an apparent sealing stress of 82 to 134 psi. Once this level was reached further service seemed to have little effect as proven in tests to 1: iry cycles.

Very few leakage problems were encountered during development. Only one case of liquid leakage was authenticated and that occurred

because a valve was inadvertently left partially open for several hours resulting in a crease across the faces of the seals. Excess leakage measured in tests with GN_2 were attributed to contamination and flaking of the Teflon during dry cycling. During a 60-day compatibility test, leakage was measured periodically after cycling until a total of 320 dry cycles and 42 wet cycles had been accumulated. A total of 168 leakage measurements were made at 50, 180, and 280 psig. All results were "zero" except in 15 cases where rates varied randomly up to 75 scc/hr; no pattern was evident.

In order to reduce the tendency of the Teflon to flake during dry cycling it was impregnated with a special lubricant (FS-5 produced by Hooker Chemical) and externally lubricated with Krytox PR240-AC grease (a product of du Pont). These lubricants substantially reduced the flaking rate.

Lunar Module Pre-Valve

This valve is used on both the ascent and descent engines of the Lunar Module providing shutoff of the pilot portion of the thruster and RCS valve isolation. Although used in a bipropellant system, the valve is used for fuel operation only .50/50 mixture of hydrazine, and UDMH). The overall valve cross section is shown in Figure 3-6. Plunger sealing and static body seals are also elastomeric o-rings. In use the valve is used in parallel redundancy to ensure that flow can always be initiated, but relying on sealing capability to limit leakage. The seal is a flat elastomeric metal seal with the soft scaling element in the poppet. the LM application ethylene propylene rubber (EPR) compound E515-8 was used. The seal is essentially a mechanically retained o-ring as shown in Figure 3-7, mating with a flat lapped metal seat.

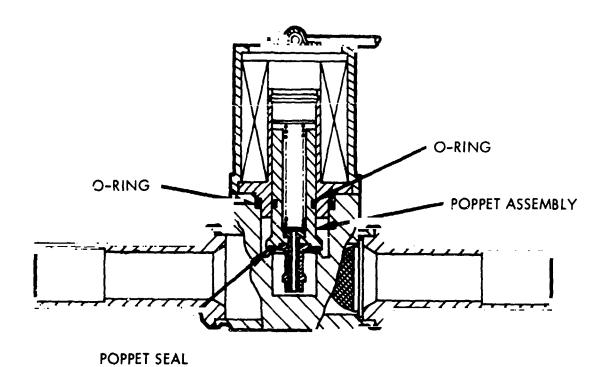


Figure 3-6. LM Pre-Valve Cross Section

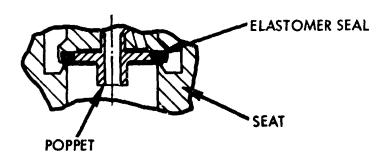


Figure 3-7. Poppet Seal Configurations
Used for LM Pre-Valve Applications

Apollo Lunar Module

 N_2O_4 and Aerozine-50 are pressurized to about 270 psia and 205 psia in the LM Descent and Ascent systems, respectively. Several different types of static, dynamic and valve seals are used to contain these propellants. All brazed tubing assemblies without static seals are used to contain the helium pressurizing gas.

In some smaller line connections such as at the pre-valve interfaces, Gamah metal-to-metal seals are used. At other engine interfaces, RACO seals (metal seal with Teflon coating) are used. Flared tube fittings, modified to accept Teflon o-rings in grooves cut in the beveled surfaces, are located at the high point bleed and low point drain. The oxidizer line trimming orifice flanges are fitted with concentric (redundant) seals, a Teflon-coated RACO seal inside and an EPR o-ring outside. Concentric seals are also used on the propellant gauging probe closures. On the oxidizer side these are Teflon o-rings (103 percent fill in MIL grooves) surrounded by formed-in-place seals of roomtemperature curing nitroso rubber injected into MIL design o-ring grooves. This ritroso material was originally compounded by Reaction Motors Division, Thiokol Chemical Corporation, and later supplied by Coast Craft Rubber Products as compounds APREL 0011 and APREL 002. On the fuel side Farker B5y1-8 butyl rubber o-rings (103 percent fill in MIL grooves) are surrounded by formed-in-place seals of a room-temperature curing butyl (Vistonex) injected into MIL design o-ring grooves. In othe locations within the oxidizer system, Teflon o-rings were backed-u the same Parker o-rings; despite the limited compatibility of butyl with N_2O_A , this technique works in static seal applications because of the limited exposure and full confinement of the outer seal. In general all static seals were assigned a leakage allowance of 1 \times 10⁻⁸ scc/second of helium at or near the operating pressure. Apparently Grumman had variable success with some of these static seals but in general the butyl o-ring seals proved reliably leak-tight.

Squib-operated valves with metal barriers serve as isolation valves within the LM circuits. The high pressure helium pressurization gas at 1750 psi in the descent stage and at 3500 psi in the ascent stage

operating temperature range is -65 to +130°F. These valves are installed in 1/2-inch and 3/8-inch lines. Allowable leakage rates past the seats is 100 scc/hour of helium at rated pressure and this specification is usually met. Seals of Teflon in the poppets are seated against a metal lands. The dynamic seals on the poppet assembly which are Teflon o-rings, have been a problem area. Initially the allowable leak rate was 20 scc/hour but this was later relaxed to 500 scc/hour because of the limited exposure time (about 12 minutes) makes the original tight specification unnecessary. Both Teflon and Parker B591-8 o-rings are used on static seals.

MC also supplied the 1/2-inch line size relief valves for the ascent stage. Integral burst discs are designed to burst at 226 to 250 psig and the valves to reseat at 225 psig. Fluid temperature range is 0 to +100°F. These valves are compatible with fuel and oxidizer vapor. Allowable leakage past the burst disc is 3 scc/hour (0 to rupture pressure) and past the valve seat is 3 scc/minute (100 to 225 psig). The poppet seat is a partially capped cylinder of Teflon which is beveled at 45° on the open end to mate with the conical metal seat; a beveled metal cyclinder, concentric to and inside the Teflon, acts as a metal stop. Teflon o-rings act as dynamic and static seals. These valves have performed well in service. A very similar relief valve, without the burst disc assembly, is used in the Bell Post-Boost Propulsion System for the Minuteman III missile.

Relief valves in the descent stage are supplied by Parker. These are installed in 5/8-inch lines and are compatibile with fuel and oxidizer. They also have a burst disc ahead of the alve. A spherically contoured metal poppet bears against the edge of a hole through the flat Teflon seat. The flexible sensing diaphragm is laminated of Teflon and Nylon. Rupture pressure for the burst disc is 288 ± 20 psig and cracking pressure for the valve is 260 psig minimum. Rated leakage past the disc is 3 scc/hour of helium up to 225 psig, and past the valve it is "0" from 248 to 50 psig.

Both the ascent and descent stages have Parker quad-redundant check

valves in the fuel and oxidizer pressurization lines (5/8-inch diameter) which are compatible with fuel and oxidizer, liquids and vapors. Operating temperature range is -65 to $\pm 160^{\circ} F$. Cracking pressure is 2.44 ± 0.44 psid overall. At any pressure differential from 0 to 270 psid the allowable internal leakage rate across the entire assembly is 3.34×10^{-2} scc/second of helium or 1.67×10^{-2} scc/second per seat. Each metal poppet is held normally-closed against the Teflon seat seal by a 1-1/2-pound spring force. These valves proved to be very contamination sensitive due to this low seat loading.

Fill connections in the LM are 1/4-inch size quick-disconnects supplied by Shultz Tool and Manufacturing Company. Teflon lipseals are used to limit the leak rate to 3 scc/hour helium or less. These disconnects were very leak-tight in service.

Apollo Service Propulsion System

 $N_2 O_4$ and A-50 are fed through stainless steel lines, with mostly welded connections, to the Aerojet AJ10-137 engine within which propellant flow is controlled by a quad-redundant ball valve built by Aerojet. Where necessary, bolted flange connections are used. These have two concentric seals, two butyl o-rings in the fuel circuit, and one Omni-Seal (Teflon U-seal with a stainless steel garter spring inside) encircled by a butyl o-ring in the oxidizer. Standard MIL grooves are used for the o-rings. Allowable leakage across these concentric seals is 1 x 10^{-7} scc/sec of helium at 175 psig. Large versions of these flange seals are used to seal the propellant tank inspection port covers.

The fill couplings, made by J. C. Carter, have Teflon o-rings and molded flat poppet seals of Teflon.

Helium, stored at 3600 psi, is contained at the joints in the 5/8-inch lines by butyl o-rings surrounded by metal vee-seals (NAR design). Gas temperatures range down to -200° F during filling and from -60 to $+140^{\circ}$ F during operation. Allowable leakage is 1 x 10^{-7} scc/sec of helium at 3600 psi. These seals have been extremely successful, only two having leaked within deliverable systems and in both cases this leakage was caused by faulty assembly.

Within the pressurization lines are quad-redundant check valves designed and manufactured by Accessory Products Company. Molded poppet seals of Resistazine 88 are fitted to the oxidizer-side valves; seals of Stillman SR634-70 are used in the fuel-side valves.

JPL/Martin Mariner '71

Nitrogen tetroxide and monomethyl hydrazine were the propellants in the Mariner '71 propulsion system. Nitrogen gas at a nominal 275 psia pressurized the tanks. Most connections were brazed but four flared tube joints sealed with Voi Shan aluminum conical seals and two tank flange joints sealed with soft aluminum crush gaskets were used. The overall subsystem external leak rate reportedly was less than 1 x 10^{-5} scc/sec of helium at 260 psig (Ref. 62). Pyrotechnic valves with solid metal barriers served to isolate the pressurant gas and propellants during the long cruise to Mars. After these valves were fired open propellants were controlled by Moog valves, and the gas by a National Waterlift pressure regulator. The poppet in the regulator is a metal ball which seats against a Teflon disc. Check valves for the pressurization lines were furnished by HTL which have a specified leak rate of 0.8 scc of nitrogen in 30 minutes. Fill valves, made by HTL, had ceramic balls seated against spherical lapped metal lands; specification leak rate was 1 x 10^{-6} scc/second of helium at 4000 psig for the gas system and at 300 psig for the fuel and oxidizer systems. Pressurant relief valves made by Calmec with integral burst discs were used to protect the circuits from overpressurization. A trapped o-ring in the poppet mates to a conical metal seat in the relief valve. The diaphragm was sealed at its edge by welding.

Reference 62 cites a number of occasions when the check valves and fill valves exceeded allowable leakage rates.

JPL/Hughes Surveyor Spacecraft

Surveyor spacecraft used mixed oxides of nitrogen (MON-10) and monomethyl hydrazine diluted with water as propellants. Engine valves were designed and built by Reaction Motors Division of Thiokol; conical seats were made of nylon for the fuel and Teflon for the oxidizer. Fill and drain valves had tungsten carbide balls seated against aluminum. Static seals were mostly MS flared tube connections.

Boeing Lunar Orbiter

Nitrogen tetroxide and Aerozine-50 were pressurized to a nominal 190 psia in the Lunar Orbiter system. Components were qualified to operate at $65 \pm 35^{\circ}F$.

Tubing assembly connections were brazed except at the engine valve interfaces where metal vee-seals were used. Pyrotechnic isolation valves with solid metal barriers were fitted to the system. Engine valves were the Marquardt R-4D model described earlier. The National Waterlift GN_2 pressure regulator had a metal ball seating against a Teflon disc. Downstream of the regulator, in each pressurization line, were quadredundant check valves built by APCO to a design essentially identical to that of the Apollo check valves (described in this section).

Agena

The Agena propulsion system uses poppet type pressure actuated poppet valves for both fuel and oxidizer service. The poppet seals for both fuel and oxidizer are conical Teflon seals machined from bar stock. This mates with a conical seat. The dynamic shaft seal on the oxidizer valve is a lipseal made of thin preformed Kel-F (mating with high surface finish chromeplated shaft).

The fuel valve dynamic shaft seal is an EPR (E515-8) o-ring on a high surface finish chromeplated shaft.

A gas generator control valve also uses the same seal designs and materials. A fuel solenoid valve (manufactured by Marotta Controls) uses o-rings, static and sliding o-ring seals as well as poppet seals made of EPR E515-8.

Check & Relief Valves

HTL Industries Incorporated manufactures a series of check valves in which the entire one-piece poppet and guiding shaft is made of Teflon or Kel-F (see Figure 3-15). These valves have been used in the MMBPS and Mariner '71. The Mariner version is fitted into 3/8-inch pressurization lines. Its specified leak rate is 0.8 scch of helium at any pressure between 1/2 and 300 psig. The configuration of the seat is a narrow land (about 7 mils wide) which seats against a flat surface of CRES. A spring load of 6 pounds plus the pressure differential across the valve, ΔP , generate an apparent seating stress, $\sigma_{\rm s}$, of approximately:

$$\sigma_s = 340 + 30 \Delta P$$

Some chattering has been experienced with this design but the seal does not seem to be noticeably damaged by that action. (For Mariner '71 a damping frictional force was designed into the valve to prevent chatter but this resulted in some valves failing to close properly until a means was developed to keep the friction force within narrow limits.) Reference 58 indicates that leakage problems were caused by contamination and swelling of the stem in N_2O_4 vapors may have contributed to sticking.

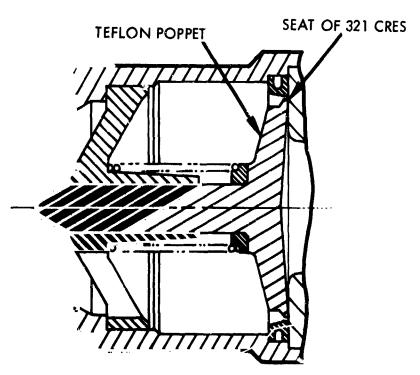


Figure 3-8. HTL Check Valve Used in Mariner '71

Apollo Check Valve

The check valve assembly used in the Apollo pressurization systems was manufactured by APCO (Accessory Products Co.). This valve is used in series-parallel assembly. It is used in A-50, N_2O_4 , and helium service. Figure 3-9 shows the valve poppet-seat configuration and the lipseal configuration. The lipseal is molded in place. The seal material is "Resistazine 88" or Carboxy Nitroso Rubber designated as AF-E-110. This check valve and seal material have also been used in the Lunar Orbiter GN_2 pressurization systems.

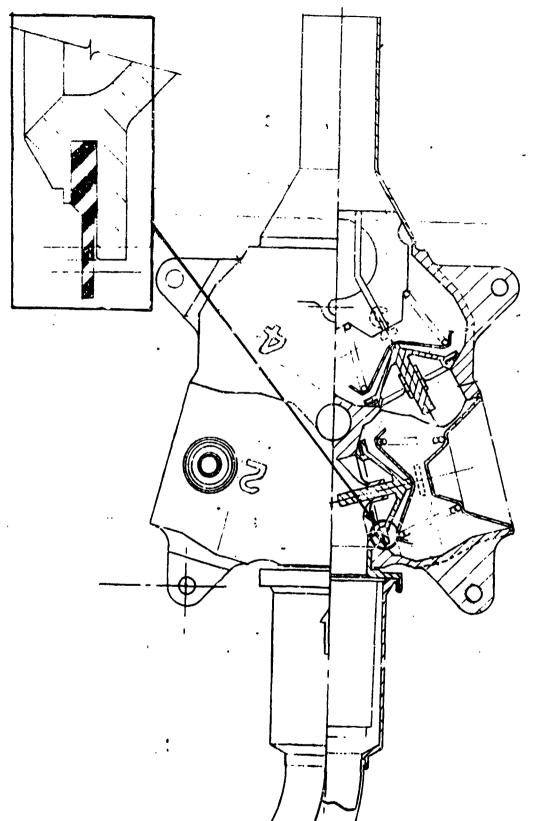


Figure 3.9. Apollo Oxidizer Check Valve Showing Seal Configuration

3.1.2 Monopropellant Hydrazine System Sealing

A brief description of seals and applications used ir selected monopropellant hydrazine systems is provided here. Information on a larger number of applications, with basic requirements was presented in Table 3-2 previously. These descriptions are designed to supplement the information in that table. These discussions are not intended to cover all the applications mentioned, but are intended to further describe typical applications and some unique sealing features.

Intelsat III, DSP, DSCS, Pioneer Valves

A common valve design is used for a number of monopropellant hydrazine engines in a thrust range of 4.1 lbf. This valve design incorporates redundant electrical coils, and series redundant seats and poppets in an integral unit. A cross section of the valve is shown in Figure 3-10. The seat-pappet configuration is a flat elastomeric seal in the poppet mating with a flat-lapped seat.

The seal is installed in the plunger and poppet and is held in position by the retainer which is crimped and electron beam welded to the plunger and poppet. Leakage across the primary poppet is detected during preliminary and acceptance tests through a test port between the seats. Sealing force is provided by the respective poppet springs plus the hydraulic force.

The poppet seal is an elastomer disk (Stillman Rubber EPT compound 724-90) and the seats are 304L and 347 stainless steel. Protusion of the seat into the seal is limited to a nominal 0.003 by a stop located in the center of the seal. Valves of this basic design are manufactured by Fairchild Industries, Farker-Hannifin Corporation and Allen Design Incorporated. The valve has passed the following qualification requirements:

Design Life:

5 years; 60,000 cycles

Temperature:

+40 to +122°F

Pressure Operating Proof

Burst

600 psig 900 psig 2400 psig

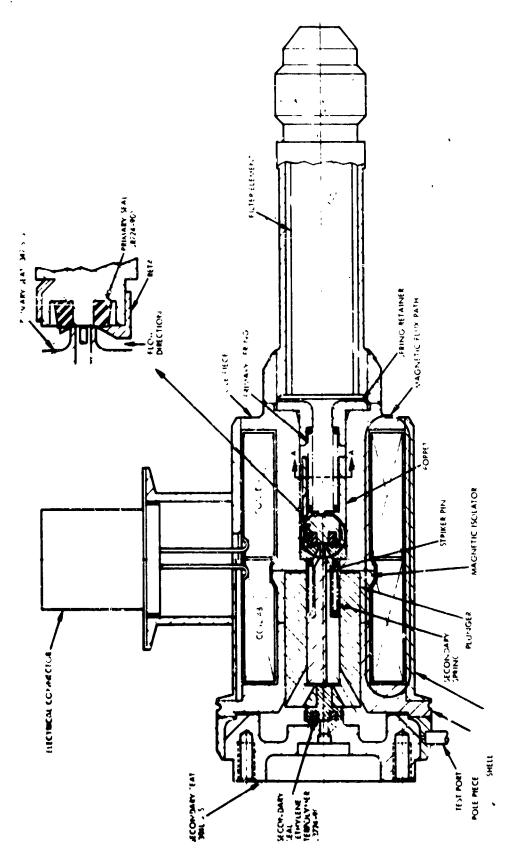


Figure 3-10. Intelsat III Type Valve Schematic

A similar configuration is also used in a gas application utilizing hydrazine decomposition products. The basic seal and seat design are similar to the Intelsat III and others described previously. The primary difference is the operating pressure which is 35 psia and cycle life requirement which is 1 million cycles.

P-95 ACS (and Similar Parker-Hannitin Corporation Monoprope Tant Valve Applications)

A number of monopropellant engine valves utilize the same basic sealing concept. These valves utilize a hard poppet-soft seat technique. (Initial P-95 AuS valves incorporated metal-to-metal seats, but are not discussed here.) The basic valve is shown schematically in Figure 3-11 although each engine application requires different housing, inlet, outlet, etc. configurations. The seal is a Teffon ring, mechanically retained in the seat and is shown schematically in Figure 3-12. The poppet contains a flat lapped sealing surface, and is a free floating manner guided by the armature. Seal load is provided by the coil spring and hydraulic force. The P-95 type valve has the following qualification requirements:

Design Life: 1 million cycles +35 to +250 °F Temperature: Pressure Operating 300 psig Proof 600 psiq Burst 3900 psig Environments Vibration 4.0 g_2^2/Hz (narrow band) 0.6 g^2/Hz (max other) 37.9 g rms Random Sine 4 g max Shock 30 g pk (max) Humidity 90% R.H.

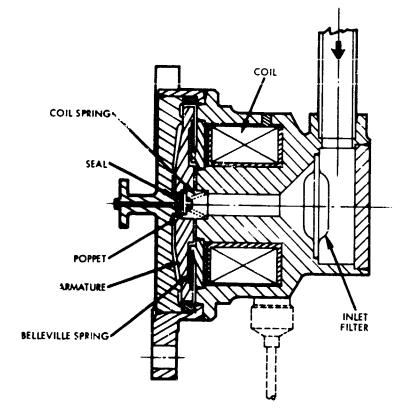


Figure 3-11. Typical Propellant Valve Cross Section

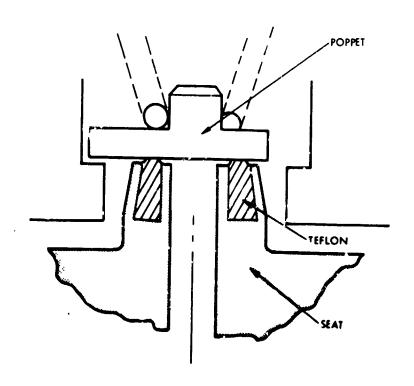


Figure 3-12. Typical P-95 ACS Valve Seat Poppet Configuration

Titan Transtage Monopropellant ACS Engine Valve

The Titan Transtage engine valves are essentially two torque motor valves in series, providing redundant shutoff capability electrically and mechanically. The sealing mechanism used is similar to that previously described for the Minuteman III PBPS axial engine. Teflon is mechanically retained in the poppet element sealing on a flat lapped soat. This valve has been qualified to the following requirements:

Cycle Life:	72,000 cycles
Temperature	+30°F to +200°F
Pressure Operating Proof	400 psig 1000 psig
Environments Vibration Random Shock	1.0 g ² /Hz 1000 g's

A coaxial solenoid valve also uses the same basic seat-poppet design. This valve is presently an alternate for the P-95 RCS propulsion system. Figure 3-13 shows a cross section view of this valve. The flexure guided armature allows the poppet to properly align with the flat lapped seat for sealing.

Intelsat IV

Two valves of interst are used on the Intelsat IV communications satellite. The thruster-valve is a series redundant metal-to-metal seat torque motor operated valve and the propellant isolation valve is a soft seat-hard poppet solenoid valve. The thruster valve is shown in section view in Figure 3-14.

The poppets are of the "Metflex" design, an HR and M proprietary name for a flexible poppet which permits alignment by rotation on a spherical support to ensure flat contact of seal and poppet. The poppet material is Kennametal and the seats Tungsten carbide with Knoop hardness in the range of 1800 and 2000. Both poppet and seat are flat lapped. The flexure tube and bias magnets provide the closing force for the downstream poppet, while the upstream poppet is spring loaded to the closed position.

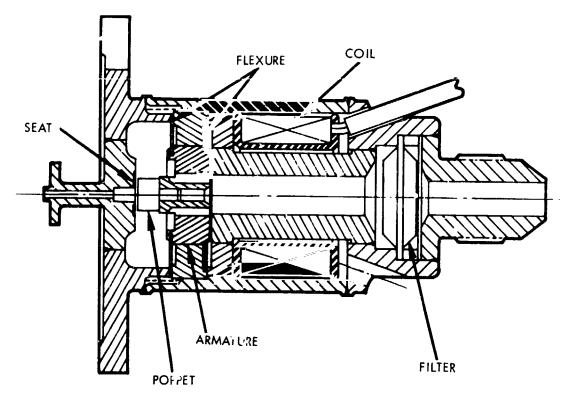


Figure 3-13. Moog Propellant Valve Cross Section

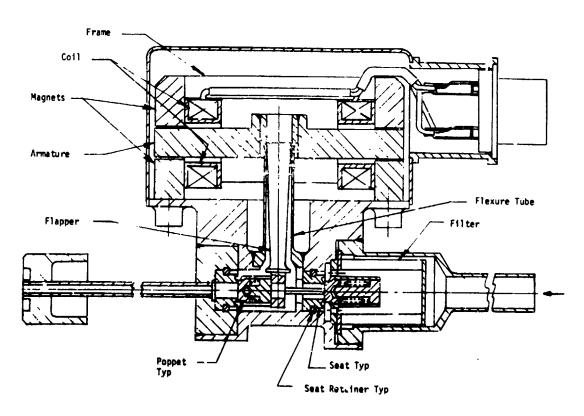


Figure 3-14. Intelsat IV Valve Cross Section

The latching solenoid valve for Intelsat IV is a pressure balanced design using bellows to accomplish pressure balancing. The soft seal material (Teflon) is mechanically retained in the poppet shown typically in Figure 3-15. Controlled seat loading is provided by the pressure balance and maximum deformation limited by the adjacent poppet metal areas. Included in the qualification requirements are the following:

Cycle Life	1,000 cycles (valve has been cycled 50,300 cycles)
Temperature	-30°F to +140°F
Pressure Operating Proof Burst	300 psig 500 psig 1200 psig
Environments Vibration Random Sine Acceleration Humidity	1.0 g ² /H⁊ (max) (25.4 g rms) 30 g peak (max) 15.5 g 95%

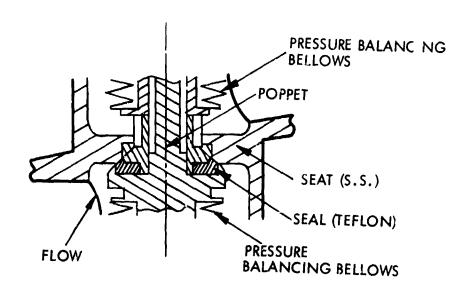


Figure 3-15. Intelsat IV Latching Valve Seal Configuration

JPL Ranger and Mariner '64, '66, and '69 Spacecraft

No primary polymeric seals were used in these monopropellant hydrazine systems. Shutoff valving consisted of pyrotechnic valves with metal sealing. The pressure regulators and fill valves used ceramic balls seated against metal. Flat crush gaskets of annealed 1100 aluminum were used at the several bolted connections which were comprised of special flanges with serrations in the sealing area.

3.1.1.3 Static Seals

Extensive use has been made of welded or brazed connections in tubing assemblies wherever disassembly is not important and high reliability joints with leak rates below 1×10^{-7} scc/sec are required. Nevertheless static seals continue to be widely used because so many separable connections are necessary (or most economical). To maximize the reliability, convenience, and cost of static seals, industry and government have expended considerable effort in searching for and testing better seal materials and configurations.

Ideal seal materials can be described but very few materials even approaching the ideal are available. The four characteristics of an ideal seal material are (1) chemical inertness in combination with propellants, (2) usability over a wide temperature range, (3) oftness or compliance to achieve good surface mating and (4) resilience or the ability to maintain a spring-like behavior so that in spite of temperature and dimensional changes, adequate load is maintained at the sealing interface. Since no such ideal material is available, most seal designs reflect the designers' attempts to overcome the shortcomings of existing materials.

Seals are fabricated in four ways: formed in place, cut from sheet, machined from stock, and molded. Formed-in-place seals are injected as a viscous liquid suspension which cures in situ. To be most practical the polymer compound must cure at room temperature. Channels must be provided for the displaced air and excess inject to escape, hence formed-in-place seals are used only as external seals or in parts to be assembled later inside other parts. Gaskets are stamped out from flat sheet stock. Final grinding or polishing of the sheet to obtain a flat smooth finish is only necessary where clamping forces are insufficient to fully yield the material. Complex seal shapes are often machined from stock, especially when small numbers are to be made from materials which can be accurately machined (e.g., Teflon). Most seals, including o-rings, Gask-O-Seals, Omni-Seals, Bal-Seals, etc., are molded.

0-Rings

The basic seal design used in storable propellant systems is the

o-ring, with the majority of o-rings being elastomeric. They are used in static, dynamic and closure application. The design, construction, and use of the o-ring are well characterized, one good source of data being the Parker Seal Company catalogue No. 5700 for elastomeric seals. Other seal companies provide design and application data for specific o-rings and applications.

As discussed earlier under the Lunar Module system, in some cases existing design data are not sufficient because of material, propellant or other reasons. In these cases the gland designs must be altered for the application. With commonly used elastomers this is relatively infrequent and MIL-Spec grooves are used in the vast majority of cases. But if the material is relatively non-elastic or subject to degradation (either chemical or mechanical, as in cold flow), then deviations may be made to assure continued sealing. The commonest deviation is to resort to a high degree of groove fill (low void); 100 to 105 percent groove fill is used with Teflon and with materials which turn cheesy or gummy after exposure. Groove cross-section shape may also be altered, shallow grooves of nearly semi-circular section being used with non-elastomers. suprisingly, many "o-rings" continue to seal in static applications if they are not opened, subjected to excess pressures, or temperature cycled, even after loosing all mechanical integrity.

Omni-Seal and Bal-Seals

A very common Teflon seal design used in a wide variety of applications is a spring-loaded U-seal which is installed so the pressure loads the inside of the seal. Typical of this class are the Omni-seals and Bal-seals. Teflon provides soft compliance at the sealing interfaces while resilience is provided by garter springs contained within the seals. Figure 3-16 shows a cross-sectional view. In some cases these seals can be used exactly as o-rings are, but they cannot be stretched very much so only glands permitting insertion without appreciable stretch are suitable.

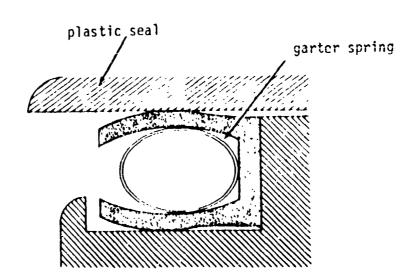


Figure 3-16. Plastic Spring-Loaded Seal

Gask-O-Seal

Another successful design using elastomers is the Gask-O-Seal device, a proprietary design of the Parker Seal Company which is also made as Still-Seals by Stillman Rubber under license from Parker. This is a metal or rigid plastic plate with elastomer seals of special crosssection molded and bonded into grooves of the desired seal pattern. Almost any size and shape of seal, regardless of complexity, can be made and proper installation is assured since the seal is fastened in the groove; it cannot pop out of non-circular grooves as can happen, sometimes unnoticed, during assembly of o-rings. Sepls can be fabricated on either one or both sides of the plate. The plate, when properly aligned with dowels or other reference surfaces, assures placement of the seals exactly where intended with no ac idental displacement during the tightening up process. Exactly the correct degree of seal compression is autimatically obtained when the plate is fully mated because it is controlled by the groove and seal cross-sections and is independent of installation procedural errors.

Polymeric Gaskets

Flat gaskets are used either against flat sealing surfaces or against serrated surfaces. High clamping forces are necessary to achieve a seal and to hold the gasket in place against fluid pressure. Sometimes tongue-in-groove designs are used.

An interesting variation on the conventional gasket design is that used in the Agena oxidizer system. These are thin Teflon gaskets confined between stepped flanges which have a series of U-type serrations on each face. This seal has been extremely successful. (Static seals or the fuel side of the Agena system are conventional EPR E515-8 o-rings.)

Metal Seals

Much of the present day technology of metal static seals was developed for rocket applications. Maintenance of resiliency over a wide temperature range and greater compatibility with propellants in comparison with elastomers were the incentives for this development. On the other hand, metal seals are more expensive and much less compliant than polymeric seals. The higher cost of metal seals is a result of the greater expense of machining metals as compared with molding polymers. As a general rule, metal seals much be machined to closer tolerances and to better finishes than required for polymer seals if reasonably low leakage rates are to be obtained. In some cases a secondary cost increment is incurred because few metal seals are reusable, due to yielding and sensitivity to scratches, whereas many elastomer seals can be reused if undamaged.

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All metal seals are either soft metals, soft-metal coated, or used so as to achieve very high sealing stresses. Soft metal flat gaskets (typically as 1100 or 1060 aluminum) clamped between serrated, raised-face flanges have seen some usage but tend to leak if temperature cycled. Spring-loading the bolts (such as with Belleville washers) has proven somewhat successful in maintaining loads as temperatures vary. Pressure-loaded U-seals of many configurations have been marketed. Most of the successful designs have the sealing surfaces plated with gold, silver, copper, Teflon, or other soft material. Perhaps the most widely used are the Harricos "S" seal and the North American Rockwell "Naflex" seals. Recently a number of metal "vee" seals have been used with success, some of these being custom designed and fabricated for specific applications by the system contractors. Low leakage rates (1 x 10⁻³ scim or less) are attained if the seals and seal mating surfaces are flat and free of scratches.

Conoseal

An industrial all-metal seal which has enjoyed considerable success is the Conoseal. This seal exploits the very high local line stresses generated at the edges of a narrow Bellevillo string when it is clamped between two beveled surfaces and forced surfaces and forced into a flatter cone angle. This stress is substantially maintained at low or high temperatures as an intrinsic property of the manner of loading. These seals require special flanges, are available to 12-inch diameter or larger, and are used both in flanged and screwed connections.

Flared Seal

Standard MS flared tubing fittings depend upon a metal-to-metal seal which tends to develop leaks at extreme temperatures, however a low-cost, soft-metal (aluminum or copper) insert is marketed by Voi-Shan which somewhat increases the Teak-tightness. This conical seal fits between the flared tube end and the conical fitting surface. Voi-Shan inserts should not be reused if reliable, leak-free service is demanded. These inserts have been used in a number of aerospace applications since the early Atlas missile system. They are standard parts at Convair-Astronautics (from whom Voi-Shan is licensed to manufacture them) and McDonnell-Douglas (St. Louis). Very low leak rates are obtained if the seal and mating surfaces are not corroded or scratched.

A number of organizations including Aeroquip, JPL, Grumman, etc., have made flared tube fittings with o-ring grooves cut into the inveled sealing surface so polymeric o-rings could be used to enhance sealing. These fittings have proven to be very reliable and leak-free provided tolerances are held. Especially important has been the improved retention of leak-tightness under vibration compared to conventional fittings.

AFRPL Bobbin Seal

An interesting metal-to-metal static seal developed for the AFRPL by the Battelle Memorial institute is the "Bobbin" seal shown in Figure 3-17. Reference 57 outlines the development of that seal. So far it has seen only limited service. One application was in the 1/4-inch, high-pressure nitrogen gas line in the MMBPS. In early tests at TRW the measured leak rates were about 1.4×10^{-4} scc/hour of helium at an average pressure of 2000 psig. Later, acceptance tests of production hardware yielded leak rates near the limit of mass spectrometer detection. This fitting was made of 347 CRES and the bobbin was made of CRES then nicke! plated. Sealing surfaces were machined to a 32μ -inch finish.

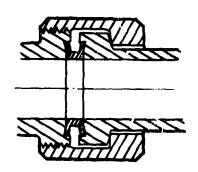


Figure 3-17. AFRPL "Bobbin" All-Motal Static Seal

3.2 Advanced Systems

Systems which are not operational or are in the development stages are discussed briefly here. Table 3-3 lists system valves in these systems and the basic attributes of these valves. As seen from the table, relatively few known systems or applications were identified. Many companies are presently working on new applications and systems, many associated with Space Shuttle, but, understandably are not in a position to divulge information because of the competitive nature of this activity. In most cases, the inference appears to be that most hardware is based on existing types of hardware and state-of-the-art sealing techniques are used. Other systems presently in manufacture, but not in the table include the Viking ACS system, Viking Lander and U.S. Com Sat. System details are not available for this report. Some of the systems are briefly discussed below.

Valve Type	Application	Fluid Yalve Supplier		System Supplier or Engine	Pressure	<u> </u>
	<u> </u>				Pstg	L
Torquemotor Poppet	ISPS Engine Value	N ₂ 0 ₄ HDA	Moog	TRW	300	(
Exterfly	Delta Engine	N ₂ 0 ₄	Post-Sea! Inc.	TRW	300	1
	Engine Valvu	A-50	Posi-Seal Inc	TRW	ļ	1
Butterfly	Titan Prevaive	N ₂ 0 ₄ A-59	Martin-Marietta	Martin-Marietta	85	4
Sel en o1d-pappet	AE Thruster Valve	N ₂ H ₄	Parker	TRW	600	Ú
Solanoij-poppet	AE Tank S/O	N ₂ N ₄	Hoog	TRW	600	0
Solenoid-soppe*	P-50 Thruster Valve	N ₂ H ₄	Farker	Hamilton-Standard	255	0
Solenoid-pappet	P-50 Thruster Valve	N ₂ H ₄	Perker	['amflton-Standard	255	0
Solenoid-pappe:	Mariner '73 Thruster Yalve	N ₂ H ₄	Marquardt	JPL	380	1
Solanoid-poppet	Marine: '75 Isolation	N ₂ H ₄	Parker	JPL		{L1

FOLDOUT FRAME

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r	Pressure	Nominal Flow Rate	Seal		Seat		Leak Rate Spec.			Cycle
	Pstg		Material	Config.	Material	Config.	Test Fluid	Test Pressure Psig	Allowable Rate SCCH	Cycle Life Req'd.
	300	0.270 0.101	Teflon TFE	Flat	304L	Flat	GN ₂	250	5.0	25,000
,	300	19.6 12.8	Teflon AF∴E-124D ∴eflon	Ring	316	Butterfly	GN ₂	300	10.0	200
-	85	4,6,7" Valve Diameter	Teflon	Ring	CRES	Butterf]y	GHe	85	3 x 10 ⁻¹⁰	1000
	600	0.015	EPT/HYSTL	Flat	304L	Flat	GN ₂	600	0.5	60,000
	600	0.02	Teflon	Flat	304L	Flat	GN ₂	600	1.0	100,000
rđ	255	0.050	Teflon TFE	Flat	304L	Flat	GN ₂	255	5.0	1.5 x 10 ⁶
,rd	255	0.0025	Teflon TFE	Flat	304L	Flat	GN ₂	255	5.0	1.5 x 10 ⁶
	380		Teflon TFE	Beveled Disc	Stellite No.6	Conical poppet	GN ₂	380	5.0	
-		(Line Size) (1.3")	Teflon TFE	Flat	304L	Flat	GN ₂	250	10.0	

Table 3.3.
Valves in Advanced or Mon-Operational System
(Bipropellant and Monopropellant)

CS

B-63 FOLDOUT FRAME

Delta Engine Valve

TRW is presently testing a Posi-Seal valve for the advanced Delta engine under contract to McDonnell-Douglas. Nitrogen tetroxide and Aerozine-50 propellants at 300 psia are to be contained over a temperature range of +25 to +225°F. The feed lines are 2.00 inches in diameter. Leakage allowances of 10 scc per hour of nitrogen and 10 and 30 psig have been specified. The total cycle life is set at 200 (wet plus dry); so far the valves in test have exceeded 1000 cycles dry successfully. A two-year storage life at -35 to +160°F is specified.

Figure 3-18 shows the seal configuration in this modified butterfly-type valve design. A Teflon ring backed by an elastomer o-ring is the main butterfly seal. (0-rings are used also to seal shafts and as static seals between the valve body and ducting.) All 0-rings are of AF-E-124D elastomer (12 used per engine).

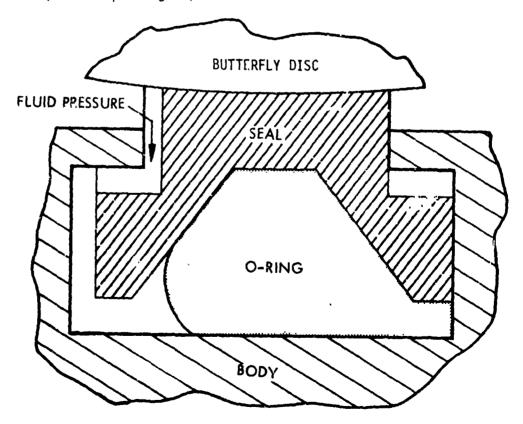


Figure 3-18 . Schematic Diagram of Posi-Seal Valve
Valve Shown in Closed and Pressurized State

Titan Pre-Valve (Ref. 68)

A new valve developed by the Martin-Marietta Corporation will replace shearable disc valves for the Titan launch vehicles. This valve is a motor operated valve in 4 in., 6 in., and 7 in. sizes. A captive Teflon seal is used as shown in Figure 3-19. A typical pre-valve (6" diameter) is shown in Figure 3-20. The Teflon is pre-stressed in the cavity by forces on the backup ring, the ring being forced against the Teflon by a threaded retainer. The stress generated by the backup ring is approximately 1000 psi; during valve closure, the butterfly is loaded against the Teflon seat by an eccentric shaft. Teflon stress in this condition is approximately 3000 psi. The seals have shown degradation during cycling, but cleanliness is important since contamination can impair the ability to seal helium. The cycle life requirement is 50 cycles, but the cycle life margin requirement (at ambient) is 1000 cycles. One unit has completed 1100 cycles successfully. The unit cycle life requirement is 20 cycles with one unit completing 20 cycles and another 200 cycles successfully. Measured leakage data at 16 psid with helium ranges from 1.06×10^{-6} to 1.91×10^{-8} on various valves in forward and reverse directions.

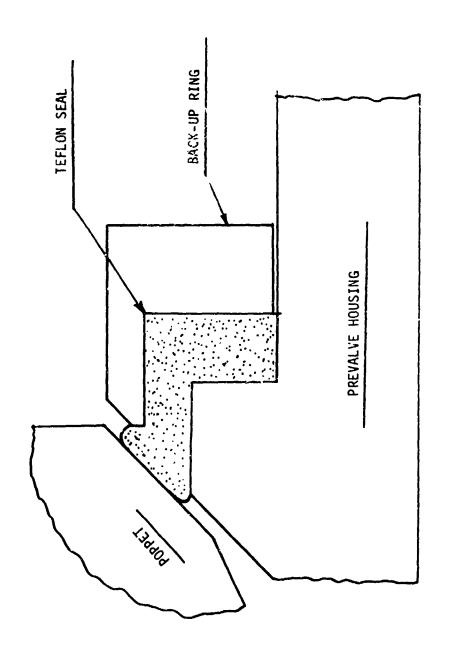


Figure 3-19. Teflon Seal & Poppet Interface MMC Prevalve



B-68

Mariner '75

A relatively large version of the Parker solenoid valve used for P-95 and other applications (effective diameter of 1.297 inches) is currently under test at JPL for use as an isolation valve in the feed-lines of the Mariner '75. This valve also uses a 12-mil wide land of flat-lapped Teflon. A net spring load of 9.5-pounds is transmitted by the poppet to the seal. Rated at 300 psig over the temperature range of +30 to +150°F, this valve is specified to leak no more than 10 scc per hour of ambient temperature helium at any pressure between 1 and 250 psig.

Atmosphere Explorer-Orbit Adjust Propulsion System (AE/OAPS)

TRW is currently designing a monopropellant system for the AE/OAPS. This is essentially an all metal system including a propellant tank with a metal diaphragm and all brazed joints. The only exceptions are the thruster valve seals which are EPT/HYSTL (AF-L-G)11) in the dual poppet solenoid valve described in Section 3.2. The tank isolation valve is a P-95 type valve of the type shown in Figure 3-13. This valve utilizes a soft poppet seal of Teflon as described in Section 3.1.

Marguardt (Classified System)

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The Marquardt Company is currently designing a monopropellant hydrazine system for a classified application. Static seals are expected to be EPR o-rings at all connections which are not welded. An exception will be at the engine valve interfaces where spring-loaded Teflon seals (either Omni or Bal) probably will be chosen. No valves have been selected at this writing but the candidates for the engine valve have captured o-ring seats.

APPENDIX C

CONTAMINATION SENSITIVITY TEST PLAN

1. PURPOSE

This plan describes a proposed test method for determining the relative sensitivity of candidate seal designs and materials to degradation in performance and integrity when exposed to fluids containing particulate contamination.

2. SCOPE

The proposed test method described herein is applicable to a wide range of fluids (inert gases, referee fluids, and certain liquid propellants, if they are compatible with the materials of construction) at any temperature suitable for the scal. Seal size should not be a limiting factor, but scaling methods would have to be developed if tests at other than full size were undertaken. Actual hardware (valves, etc.) can be used as the test fixture, provided the test replica can be installed in place of the hard seating surface and provided that acceptable control of such variables as concentricity, etc., can be maintained; otherwise a precision test fixture designed to accept the test replica and test seals is recommended.

3. INTRODUCTION AND BACKGROUND

Development of seals for fluid control devices such as valves, regulators, and so forth, has been largely an empirical process. Few purely analytical approaches to design and verification of performance, life, or reliability have been successful. This is especially true of contamination sensitivity assessment.

Sensitivity of a seal to the presence of contamination in the fluid being controlled is evidenced by leakage, wear or other damage, and jamming. The ideal sensitivity assessment method would allow competing candidate seals (and seal materials) to be evaluated for relative immunity to the presence of particulate contamination. (Non-particulate contamination, such as greases, moisture, etc., are not considered in this document.) It would perform the evaluation under controlled

conditions so a direct correlation could be obtained between degree of degradation and the pertinent parameter values (seal design, material properties, operating conditions, particle properties, etc.).

Most of the methods used in the past have involved a significant number of unknowns. Typically a fluid containing some artificially induced contamination is flowed through or past the seal under test, and the wear and leakage are measured after a certain time of exposure has elapsed or a number of operating cycles have been performed. Contact between the particles and the seals is by chance and usually undetected except by inference from the damage observed after the test. The particle population may be entirely random (typically, Arizona Road Dust, in which case correlation with particle size and shape is impossible) or a narrow cut of sizes (typically, spheres of which a high percentage fall within a radius of each other in size, in which case the results are of limited meaning).

4. TECHNICAL DISCUSSION

4.1 Particle Descriptions

Particles found in aerospace fluid components are usually comprised of:

- Metal compounds (rust and other corrosion products, grinding wheel dust, weld slag, lapping compounds)
- Carbon (smoke deposits, combustion products)
- Earth substances (silicon dioxide, glass, diamond dust, etc.)
- Metals (machining chips and shavings, slivers and particles broken, extruded or worn from rubbing or fatigued surfaces, plating, solder, weld splatter, braze droplets, etc.)
- Polymers (slivers, flakes and shreds cut, extruded, or worn from plastic and rubber seal, hoses, diaphragms, etc.)
- Protein (hair, flakes of skin, insects, plant segments)
- Crystals (non-volatile residues from solvents, etc.)
- Fibers (cotton, wool, nylon, paper, wood, etc.).

Particles come in a wide range of sizes, shapes, and hardnesses. In aerospace fluid system practice there is seldom any concern about particles below 5 mlcrons (µ) in largest domension. Figure C-1 shows

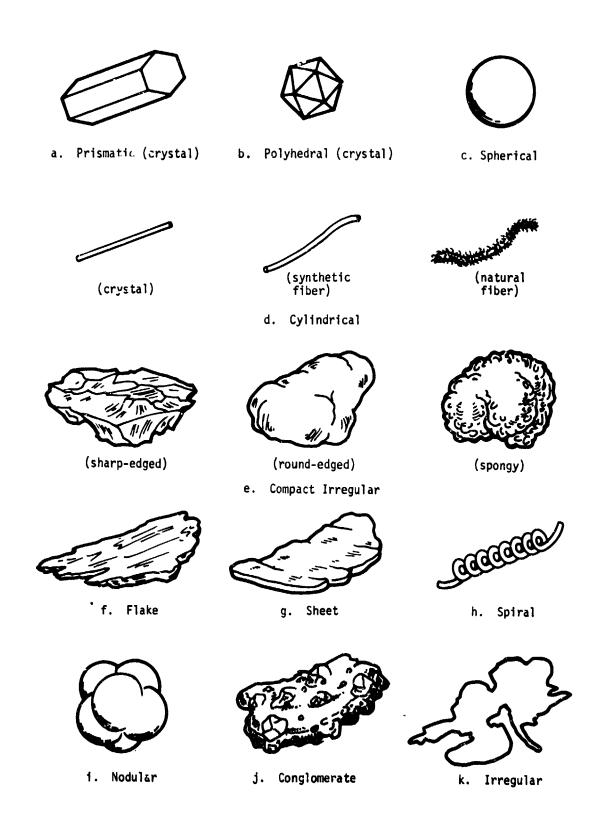


Figure C.1. Classes of Particle Shapes

the different classes of particle shapes usually encountered. It is to be recognized, of course, that these are arbitrary divisions, and real particle populations show infinite variety which results in continuous shadings of shapes from class to class. Hardness ranges from the very hardest known substances (diamond) to very soft (greases and oils) with which this discussion is not concerned.

Metallic corrosion products are crystalline in form and tend to grow to large sizes if undisturbed; many are very hard compared to polymeric seals but frangible. Rust, especially, is easily crushed or abraded into tiny particles so that the preponderance of the population (in terms of numbers) is small (below 10μ). The larger metallic compound particles which form compact crystals may appear to have had the corners broken off. Those compounds which form into a needle-like crystalline structure usually are broken into segments if worked.

Carbon tends to be deposited out as tiny, soft clumps of the amorphous form but can appear as hard, rounded nodules.

Earth substances also tend to appear as compact shapes with edges largely broken off, but the size range is very wide and the frequency of occurrence falls into the log-normal category (many more small particles compared to large particles). Glass is an exception since most glass is newly formed and has not been finely ground. Glass particles are often sharp edged and the shapes include needle-like and flake-like pieces as well as the compact irregular ones. Diamond dust and carbides (from tools or grinding operations) are generally comprised of large-sized particles of irregular, compact shape. Earth substances are almost all very hard.

Metal particle populations may not be skewed so strongly to the small size end of the spectrum. The size and shape of metal particles are strongly influenced by the particular metal and the manner in which they were generated. Larger machining chips and shavings have their own characteristic shapes (usually flat or spiral), which are broken down to charp-edged compact irregular particles. Large pieces of plating come off as this sheet-like pieces, which may be broken down to compact particles. Wear particles tend to be smaller; the shape depends on the kind of wear mechanism which is active (adhesive wear and plowing

creating very irregular to fibre-like particles, while abrasive wear results in more nodular shapes of spongy agglomerated smaller particles). Solder, weld splatter, and braze droplets tend to be large and rounded to spherical (due to the surface tension forces.) Metals are hard compared to polymeric seals.

Polymer particles also tend to be large, irregular, and of the same range of hardness as polymeric seals. Rubbers, especially, tend to form stringy ligaments or loosely connected fragments, except when the core is filler particles; these smaller particles tend 'a compact irregular.

Skin flakes appear in all sizes, but hair and insect parts tend to be large. Plant segments are of irregular shapes although some are close to being spheres or nodules (spores, pollen, etc.) These protein particles are also in the same range of hardness as seal materials.

Crystals deposited out upon the evaporation of solvents, etc., cover the full range of size and hardness and tend to be frangible.

Fiber particles tend to be long and either straight or curved, but fragments of wood are clumps of fibers and so often are close to prismatic or sharp-edged irregular in shape. Fibers usually are somewhat harder than the softer seal materials.

Not all particles are of a simple substance. Composite particles frequently are seen, especially if several mechanisms are operating simultaneously to generate particles, and one or more of them produces a substance which tends to form a matrix.

Table C-1 summarizes the more common shapes encountered for each material and mode of generation.

4,2 Particle Origins

Particles are either generated within the components or are carried into them. Some particles remain after post-marifacture and assembly cleaning. Others are carried in by the fluids or may enter during periods when ports are open. Particles are generated by corresion, wear, extrusion, etc. Design, manufacturing precision of the

Table C-1. Comparison of Particle Shapes and Mode of Generation

Irregular	×		×	 	×		×	×	<u> </u>					(×	×		_					Ţ.	
Conglomerate [1]	×	×			×											Ì		<u> </u>					
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Mode of Generation	Corrosion	Grinding and Lapping dust	bold slay	Smoke from	Combustion residue	Divided rock (send, clay, etc.)	Glass fragmits	Diamond dust and carbides from	Machining chips and shavings		Plating loss	Solder, weld and braze splatter	Cutting	Extrusion	Hear	Frie falleut	Skin flaking	Insect	Plant segmentation	fallout	Synthetic fiber	Paper fallout	Mood fracture
Materiai	3	2 5 2 5 2 5 2 5 2 5 2 5 2 5 2 5 2 5 3		Fras				Substances			Netals			Polymers			Santa da	-			Fibers		

components' moving parts, and the techniques used in assembly, adjustment and storage play major roles in determining how many and what kinds of particles are generated.

4.3 Role of Particles in Degrading the Performance and Life Expectancy of Seals

Particles increase seal leakage reces by reducing the conformation of the seal to the surface. They do this by mechanically separating the seal from the surface or by damaging either the seal or the surface. When hard particles are trapped between soft seals and hard surfaces, the seal is indented so a flow path is created adjacent to the particle. Under high loads or repeated loading, the indentation may become deep enough for the particles to become imbedded in the seal. Permanent (non-recoverable) damage caused he particles falls into the categories of cutting, scratching and wear. Usually only the soft seal is cut, but either the seal or surface may be scratched or worn. Erosive wear is a special case where the damage is caused by impingement of a high velocity fluid stream containing particles. Some seals which act by scrubbing or other motion of close fitting parts may become jammed if large particles become wedged between the moving members.

4.4 Sensitivity Relations

The degree to which a seal is sensitive to particles depends on a complex interaction of a group of parameters including:

- Seal material hardness and cut growth properties (at the temperature of contact)
- Seal design (width of land, redundancy of sealing, etc.)
- Particle hardness, shape and size ratio to critical seal dimension-typically land width)
- Load (stress level)
- Repetition of contact
- Fluid flow conditions near and over seal (as it affects recirculation or deposition of particles)
- Scrubbing motions.

1 1

5. METHOD

The following paragraphs describe the proposed methodology for characterizing particles, subjecting seals to the effect of these particles, and measuring the resultant leakage and damage. An empirical approach is utilized, as contrasted with analytical procedures. The intention is to provide a method which is suitable for evaluating a single seal, alternative materials for use in a given seal design, or competing candidate seal designs. Mechanical effects only are evaluated, no including erosion by impingement. Chemical reactions are not measured.

Basically the method involves the creation of permanent contours on the hard mating surface (against which the seal bears) which are mechanically equivalent in size, shape, an position, to hard (incompressible) particles which might become trapped on the surface during closure. Since the contour is an integral part of the seat and unchanged by repeated closure, the variables of particle size, shape, and location, * Figure C-2 graphically summarizes this proposed technique. (Previous experiments have attempted to achieve these same ends by glueing particles on seats, but the impermanency of such an arrangement was a major flaw in the method.) By creating a set of hard metal replicas which can be directly substituted for the seat in a test fixture, any number of candidate seals can be subjected to the mechanical presence of exactly the same particle shape and location. After repeated closing of the seal against the replicas, the leakage increase and wear of each seal can be evaluated and directly correlated with the known and controlled particle conditions represented by the replica countour.

5.1 Characterization of Representative Particles

Since the hardest particles represent the "worst case," the only variables needed to characterize particles are size and shape.

Size is a primary variable of interest and can be handled as a ratio of some critical seal dimensions (notably land width and thickness for

^{*}Note: Although this procedure is aimed at evaluation of contamination effects the basic method is equally suitable for evaluating the effects of seat conditions (scratches, etc.) on seal performance and life.

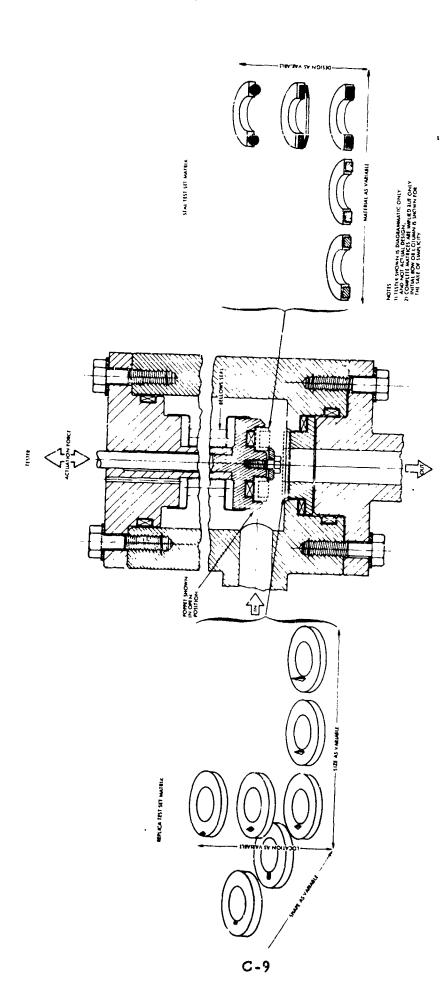


Figure C. 2 Diagrammatic Representation of Contamination Sensitivity Testing Scheme

circular seals of rectangular cross-section). Probably three size ratios would be sufficient to characterize the response of seals to a particle population; only the larger size ratios are significant since very small particles have little influence on compliant seal leakage or integrity.

Reducing the infinite array of real particle shapes down to a set of representative shapes which is small enough to be testable presents something of challenge. It could be argued that a minimum set is comprised of two worst cases: long cylinders (fibers) and an acute 3-sided pyramid. The long cylinder represents all the fibers and long particles which can bridge across seal lands, and the pyramid all the thick, sharpedged particles which tend to indent, cut and wear seals. Although this narrow representation is probably too severe a limitation to be realistic, it does seem to present the worst cases in a set which is easily testable.

It is recommended that consideration be given to analysis of actual particle samples, if this is practicable, results of which can be used to choose a more representative test set population if so desired.

5.2 Replication

To have an unchanging surface contour representing the presence of a particle on the hard seating surface, it is necessary to fabricate a replica of the surface. Fabrication of such replicas is yet to be demonstrated but at least one of the following methods should prove feasible:

- Electroless plating
- Sputtering
- · Die casting.

The major problems to be surmounted are the small size of typical contamination particles and the tendency of the particles to move during replication. Replicas should be made of tough, wear-resistent materials such as chromium, nickle, or similar metals.

5.3 Test Setup

The test setup is comprised of a test fixture, actuator, measuring equipment, and test sets. Basically the test fixture shown in Figure C-2 is a heavy-duty poppet valve but ball valves, butterfly valves, etc., could be used. The poppet seals and seat are interchangeable with any

of the test set seals and seat replicas. Test fluid is introduced via the inlet port and exhausts, when the poppet is off the seat, via the outlet. With the poppet closed, seat leakage is measurable at the outlet if a suitable leakage detector is connected; for large leak rates this could be a bubble collector and for low leak rates it could be a tracer gas mass spectrometer (sniffer). An appropriate actuator connected to the actuation shaft is necessary to cycle the poppet.

Figure C-3 shows a recommended setup for testing. In this case it is assumed that the test fluid will be an inert gas, preferrably helium. Alternatively, nitrogen or other gas or liquid (including propellant) could be used as the test fluid.

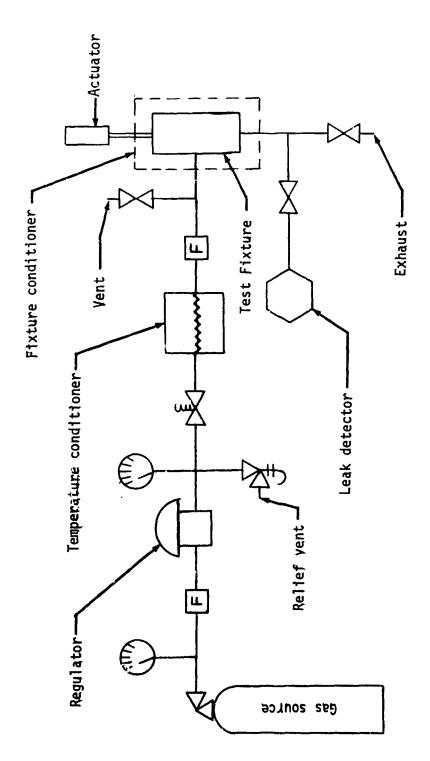
Containment of the test fixture within a conditioning chamber is indicated. This would permit controlling the ambient temperature (and pressure, if desired) of the fixture. A temperature conditioner in the inlet line is for the purpose of the test fluid temperature. A very good filter, rated well below the test particle size, should be installed in the inlet line to be sure actual particulate matter is excluded from the seat since such matter might cause leakage and/or damage which would invalidate the test data.

Not shown but essential to wear rate measurement is a precision scale and microphotography equipment.

5.4 Testing

Five potential factors in the test matrix (seat design, seat material, particle shape, particle size, and particle location) have been mentioned above. In addition, there are a number of other potential factors such as temperature, load, rate of closure, test fluid, and amount of scrubbing motion. The total test matrix design must be derived with consideration for the schedule, cost, and other programmatic factors.

Generally, it turns out that an optimum test program starts with a series of screening tests to eliminate the least worthy designs as quickly as possible. These screening tests usually are done at some relatively severe combination of conditions; otherwise, not enough candidates may be eliminated. (In some cases, however, there are few candidates to start with so all may be subjected to the full matrix of conditions.) One



Note: Precision scale and microphotography equipment needed to measure seal wear

Figure C-3. Contamination Sensitivity Test Setup Using Gas as Test Fluid

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step in designing the screening test program to minimize its magnitude is the establishment of whatever mandatory performance goals are deemed necessary. Seals which fail to achieve these "go/no-go" standards are dropped from further testing immediately. Usually these are relatively straightforward standards such as an upper limit on leak rate at prescribed conditions of pressure and temperature or a certain amount of wear. To make these standards most effective in minimizing test effort some compromise must be struck in the frequency of making leakage and wear measurements. If these measurements are time consuming, it may be most economical to take them at infrequent intervals, accepting the fact that extra tests might be made beyond the necessary minimum at which the go/no-go standards were exceeded. Conversely, if measurements are quickly made (or continuously made as might be the case with leak rate), and cycling the fixture is laborious, then frequent checks of performance may be best. Only the specific circumstances of a given test series can be used as guidance in optimizing the testing schedule.

Two kinds of test programs are common. One seeks to learn the ultimate capabilities of the candidates. This type of program may be very long and expensive if good seals are available since each candidate is tested for as many cycles as it takes to degrade it below the set standard of performance. The other measures whether the candidates can meet the arbitrary goals of a specified number of cycles which may be set far short of the candidates' ultimate capabilities; this is often a shorter program since each seal is tested only until it fails or reaches the goal.

Still another dimension in designing the test matrix is the "mix" of cycles at each value of the test conditions. In some cases there may be a simulated mission profile in which the proportion of cycles at each condition (of temperature, for instance), and the length of the open and closed periods, is the same as anticipated in flight. At other times there may be some mathematical basis in statistical analysis for choosing the cycle distribution. Or, as mentioned above, everything may be set at the "worst case."

Performance of the tests can be expected to be straightforward with little technical difficulty. Depending upon the method chosen, leak

rate measurement may be somewhat problematical unless the downstream side of the seat is fully vented (or a good vacuum drawn) after the closure portion of the previous cycle. No highly permeable materials should be used in the downstream circuit if very high resolution measurements are to be made since such permeable materials will outgas for some time after closure.

Seal materials conform so leak rate will gradually decay as the seal conforms slowly to the seat. This may be somewhat offset by a tendency for an increase in leakage with materials which undergo a substantial stress relaxation in designs with limited deformation (stroke bottoms out against metal stops). These minute variations in leak rate will not be observed unless the measurements are very sensitive and prolonged.

5.5 Data

fest data include descriptions of the matrix test conditions (summarized in Table C-2, the schedule of cycles performed, and the measured wear and leak rates.

Table C-2. Matrix Parameters to be Recorded as Part of the Test Data

Seal design Seal material Scrubbing length Concentricity error Limit of stroke Temperature of seal Test fluid Test fluid pressure (upstream) Test fluid pressure (downstream when open) Test fluid temperature Test fluid flow rate (full-open position) Closure rate or program Equilibrium load (stress) Cycle description: time held open time held closed

Replica: Shape

Shape Size Location Leak rate data usually require little analysis except perhaps some smoothing and change of units, but wear data are hard to gather, correlate and interpret.

Wear is expressed as weight or volume removed, or change in dimension. Wear tests are often made under simulated use conditions, i.e., while the seal is immersed in the fluid to be contained in service. Weight, volume, and dimensional change data taken under these circumstances on polymer seals should always be suspect if the seals are prone to swelling or shrinking during liquid immersion and evaporative loss in vacuum. Removal of a permeable seal from a inert, high pressure gas test may result in "ballooning" unless the pressure reduction period is sufficiently long for the gas to escape. Corrections of such wear data for swell, shrinkage, sublimation, and ballooning are difficult to achieve unless separate test have provided data on these factors with all pertinent test conditions clearly defined as being identical to those during the wear tests. Usually this is best acquired on the same specimens which are to be subjected to wear testing in order to eliminate errors due to differences from specimen to specimen. Otherwise, the wear test becomes in actuality a "combined effects" test from which the data yield a totalized number expressing dimensional weight changes due to swell (or shrinkage) and wear as inseperable components.

Surface contour changes are another form of data which may be taken. Generally this is in terms of the surface finish expressed as RMS height in micro-inches. A profilometer or similar device is used for harder surfaces but soft rubbery materials may present a problem where surface measurements are impractical, optical methods may be used to judge how much wear has changed the surface contours. Currently the very best techniques are light scattering and Scanning Electron Microscope (SEM) photographs. Very subtle changes can be detected by those means but they are basically qualitative rather than quantitative.

APPENDIX D

Bibliography

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- 221. Blackburn, J. F., et al, (editors), "Fluid Power Control", The M.I.T. Press, Cambridge, 1960.
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- 223. Chu, H. N., et al, Final Report, "Development of Efficiency and Life of Expulsion Bladders", Report R-7391, Rocketdyne, a Division of North American Rockwell Corporation, Canoga Park, 29 February 1968.
- 224. Manson, S. S., "Behavior of Materials Under Conditions of Thermal Stress, Report 1170, National Advisory Committee for Aeronuatics. Washington, 1954.

APPENDIX E

MATERIAL SPECIFICATIONS

This Appendix provides preliminary Material Specifications for the recommended Space Shuttle Seal Materials AF-E-411 and AF-E-124D.

The purpose of these specifications is to allow procurement of these materials, with assurance of material consistency. Since a finalized material specification must take into account all the factors in fabrication and compounding, additional investigation is required to ensure that a realistic specification results. This effort is being conducted under Air Force Contract, by TRW Systems.

At present, TRW Systems is the sole supplier of AF-E-411 and AF-E-124D materials. Materials obtained through TRW Systems will conform to these preliminary specifications.

APPENDIX E-1

MATERIAL SPECIFICATION (PRELIMINARY) -

RUBBER, EPDM COMPOSITE

DESIGNATION: AF-E-411

1. SCOPE

- 1.1 <u>Scope</u>. This material specification establishes the requirements for an ethylene-propylene terpolymer (EPDM) elastomer compound (see 6.3) intended specifically for seals.
 - 1.2 Classification. The designation of this material is AF-E-411.

2. APPLICABLE DOCUMENTS

SPECIFICATIONS

Federal

0-A-51

Acetone, Technical

TT-I-735 (C406426)

Isopropyl Alcohol

Military

MIL-P-26536

Propellant, Hydrazine

OTHER PUBLICATIONS

American Society for Testing and Materials

ASTM D 15	Compound and Sample Preparation for Physical Testing of Rubber Products
ASTM D 395	Compression Set of Vulcanized Rubber, Tests for
ASTM D 412	Tension Testing of Vulcanized Rubber
ASTM D 624	Tear Resistance of Vulcanized Rubber, Tests for
ASTM D 1460	Change in Length of an Elastomeric Vulcanizate Resulting from Immersion in a Liquid, Test for

American Society for Testing and Materials (Cont'd)

ASTM D 1708

Tensile Properties of Plastics by Use of Microtensile Specimens, Test for

ASTM D 2240

Indentation Hardness of Rubber and Plastics by Means of a Durometer, Test for

3. REQUIREMENTS

- 3.1 Material. The material shall be ethylene-propylene terpolymer elastomer formulated and processed to meet the requirements of this specification. All materials shall be of high quality and suitable for the purpose intended and free of impurities reactive with hydrazine.
- 3.2 <u>Composition & Physical Properties</u>. The composition and physical properties shall conform to the requirements specified in Tables I and II.

Table I. Formulation and Cure

Component	Parts by Weight	
Nordel 1635, duPont	100.0	
Cab-O-Sil M-5, Cabot Corp.	25.0	
8-3000 Resin, Dynachem Corp.	25.0	
Teflon Powder T-8A, duPont	5.0	
Zinc Oxide, Baker Reagert	5.0	
Calcium Oxide, Baker Reagent	5.0	
Vinyl Silane A-172, Union Carbide	1.0	
D1-Cup R, Hercules	2.0	
Total Parts -	168.0	
Cure - 30 Min/350°F		
Postcure - 2 hours/225°F		

Table II. Physical Properties

Property	Requirement
Original:	
Tensile strength, psi, min.	2100
Elongation, %, min.	170 ± 25
Hardness, Type A durometer	88 ± 5
Tear strength, 1b/inch, min.	200
Following exposure to conditions as specified:	
Compression set, 22 hours at 160F,	
24-hour rest, % max.	25
Fluid resistance, hydrazine immersions,	
96 hours at 160F:	
Compatibility, pressure rise, psi, max at	
160F (greater than control container)	5
Volume increase, % max. (measured wet at 75F)	5
Tensile strength, % of original value (at 75F)	75-125
Elongation, % of original value (at 75F)	75-125

- 3.3 <u>Workmanship</u>. Each molding shall be free of functional defects, foreign materials, and shall be uniform in quality; it shall also be free from mold flash unless otherwise permitted by the specific test method. There shall be no visible evidence of surface irregularities, such as unbonded sections, excessive holes or dents, blisters, and pits greater than 0.005 inch in height or depth. Small surface blemishes less than 0.005 inch in height or depth shall not be allowed when grouped closely together and cover more than .3 percent of the total surface area.
- 3.3.1 <u>P.gmentation</u>. Markings on the rubber surface which appear to be two dimensional (i.e., appear to be a discoloration rather than an inclusion) shall be acceptable as long as the following criteria are met:
 - (a) The discolored portion does not affect the flexed morphology of the rubber.
 - (5) The discolored portion does not react with hydrazine same compatibility requirement as in Table I.
 - (c) The discolored portion does not show any tendency, upon repeated flexing, of separating from the rubber substrate.

4. OUALITY ASSURANCE PROVISIONS

- 4.1 Qualification. Materials supplied to this specification shall have been qualified in accordance with all of the requirements of this specification.
- 4.1.1 Qualification Certification. The supplier submitting material for qualification shall provide certification that the materials submitted conform to all of the requirements of this specification and represent normal production run material.
- 4.1.2 Qualification Sampling. Qualification sampling shall include two test slabs prepared in accordance with the requirements of ASTM D 15 and two molded compression set buttons prepared in accordance with ASTM D 395, Type I.

CAUTION: No marking small be affixed or applied to a sample, only to the package in which it is contained. (See Section 5 for cleaning, packaging, and marking requirements.)

- 4.1.3 Qualification Testing. Qualification testing shall consist of demonstration of conformance to all of the requirements of this specification.
- 4.1.4 Qualified Status and Requalification. A material that has been qualified in accordance with this specification require requalification for any change of raw materials and/or processing from that used for qualification.

4.2 Acceptance.

- 4.2.1 Acceptance Certification. The supplier of material shall certify that each shipment of material made to this specification conforms to all of the requirements of this specification. Certification shall include the actual results of laboratory tests to determine conformance.
 - 4.2.2 Acceptance Sampling.
- 4.2.2.1 <u>Batch</u>. A batch shall be the quantity of material compounded on a mill at one time.
- 4.2.2.2 <u>Preproduction Sampling</u>. Preproduction sampling shall include from each batch two test slabs prepared in accordance with the requirements of ASTM D 15 and two molded compression set buttons prepared in accordance with ASTM D 395, Type I.
- 4.2.2.3 <u>Production Sampling</u>. Production sampling shall include each production item.
 - CAUTION: No marking shall be affixed or applied to a sample or to a molded part, only to the package in which the item is contained. (See Section 5 for cleaning, packaging, and marking requirements)
 - 4.2.4 Acceptance Testing.

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4.2.4.1 Preproduction Testing. Prior to the start of a production run for each batch of material the preproduction samples shall be tested to all of the requirements of this specification.

- 4.2.4.2 <u>Production Testing</u>. Each production item and subsequently the packaging for the item shall be inspected to determine conformance with the requirements of 3.3 and Section 5, respectively.
- 4.2.5 Rejection. Sample material and production items failing to meet the test requirements in 4.2.4 shall be rejected.
- 4.3 <u>Test Methods</u>. Qualification and acceptance testing of the material to the requirements of this specification shall be in accordance with the following:
- 4.3.1 Tensile Strength and Elongation. The tensile strength and ultimate elongation shall be determined in accordance with the requirements of ASTM D 412, using Die "D" for the specimens. Results shall conform to the requirements of Table I.
- 4.3.2 <u>Hardness</u>. The hardness shall be determined in accordance with the requirements of ASTM D 2240. Results shall conform to the requirements of Table 1.
- 4.3.3 Tear Strength. The tear strength shall be determined in accordance with the requirements of ASTM D 624, using Die "B". Results shall conform to the requirements of Table I.
- 4.3.4 Compression Set. The compression set shall be determined in accordance with the requirements of ASTM D 395, Method B at the aging time and temperature specified in Table I. Results shall conform to the requirements of Table I.
- 4.3.5 <u>Fluid Resistance</u>. The procedure outlined below is used to determine the fluid resistance and the relative compatibility of the rubber specimens with propellant-grade hydrazine (See 6.4).
 - (a) Cut three specimens of the rubber from a test slab, using the ASTM D 1708 die.
 - (b) Clean the specimens using in sequence a detergent solution, 0-A-51 acetone, and finally reagent grade isopropyl alcohol. Dry for 5 minutes at ambient temperature in a dirt/dust-free environment. Do not handle with bare hands.
 - (c) Measure the length and weight of each specimen.

- (d) Using rubber gloves which have been pre-rinsed in isopropyl alcohol and dried, place the rubber specimens into the bottom of an 85 cc glass Aerosil Compatibility Tube (e.g. VWR Scientific Company Catalog No. 62810-000) precleaned and dried as in (b) above. The tube must have at least a 100 psig safety rating. The primary cap seal must be Teflon and the secondary seal shall be cut from the test slab.
- (e) Add slowly 50 ml of MIL-P-26536 hydrazine to each tube.

<u>WARNING</u>: This step must be performed with care by a propellant handler; hydrazine is a dangerous chemical.

(f) The Aerosil Compatibility Tube shall be fitted with all 304 CRES fixtures, tubing, gauges, and valves as follows:

To the threaded flange cap attach a "T" fitting. From one leg of the "T" attach a 1/8 inch tubing (See (g) below) leading to a compound Bourdon-type pressure gauge (calibrated in inches of Hg on the vacuum side and in psia on the pressure side; a convenient size is 0 to 100 psia).

CAUTION: Do not use bulkhead fittings to interface from pipe threads to other types such as AN, because bulkhead fittings require 0-rings for sealing. Fitting threads shall be wrapped with TFE pipe tape prior to assembly. The whole assembly, prior to fitting on the tube, shall be rinsed in isopropyl alcohol and dried (See (b) above).

(g) Place the Aerosil Compatibility Tube and the valve inside an oven at room temperature. The oven shall have been precalibrated to the desired temperature (See Table I). Pass the 1/8 inch tubing (see (f) above) through the oven wall so that the pressure gauge can be maintained near ambient temperature. The total volume of the 85 cc tube, fittings, gauge, valve, and tubing should be approximately 95 cc.

(h) Place the oven in a bunker or test cell which has a viewing window through which the pressure gauge may be observed.

<u>WARNING</u>: A power switch for the oven shall be placed outside the test bay in order to turn off the oven if the pressure approaches the safe limit of the glass compatibility tube. If this occurs, the oven must be turned off immediately inasmuch as excessive pressure can be quite dangerous.

- (i) Connect the valve to a vacuum pump isolated from the hydrazine by a cryogenic trap (CRES or glass tubing and fittings from the valve to the trap) and evacuate the apparatus for several minutes, or until the hydrazine ceases to bubble (outgassing has ceased). The compound gauge must indicate a vacuum of at least 28 inches of Hg. Close the valve and disconnect the vacuum pump.
- (j) Check the system for zero leakage by reading the compound gauge after 30 minutes. Eliminate any leakage. When the system is leak-free, turn on the oven to the calibrated temperature and when the oven reaches the desired temperature, start the storage time.
- (k) At the end of the required storage time (or before, see (h) above) record the pressure and turn off the oven. Cool to ambient temperature (at least several hours), record the pressure again and vent the valve to relieve any residual pressure.
- (1) In a well-vented fume hood remove the flange cap, decant the propellant and, while still working in the fume hood, place the specimens on laboratory wiping tissues (Kimwipes, Kimberly-Clark Company, are satisfactory) and carefully blot dry. Then remove the specimens, using the same clean gloves described in (d) above.

WARNING: Do not use a sulfite-type paper for blotting inasmuch as this paper in contact with hydrazine is a fire hazard.

- (m) Measure the blotted samples for length and weight. Calculate the length and weight change. The volume change may be calculated from the length change in accordance with ASTM D 1460.
- (n) In a clean, pre-weighed round bottom flask carefully evaporate to dryness an aliquot of the decanted propellant and then calculate the propellant's non-volatile residue in milligrams per 100 milliliters (mg/100 ml) as the units.

<u>WARNING</u>: This step must be done carefully and in a safe place by a propellant handler; the procedure can be very dangerous.

- (o) A control propellant sample shall be processed in accordance with Steps (d) through (n) exactly as above except that the rubber specimens shall not be introduced.
- (p) The non-volatile residue from Step (o) shall be compared with that from Step (m) (with the rubber specimens). See 6.5.
- (q) Allow the exposed rubber specimens (from Step (m) above) to air-dry for 24 hours and then measure and test them for the properties specified in Table I. The results shall conform to the requirements of Table I.

The results of this test shall conform to the requirements of Table I and also the compatibility portion shall conform to the requirements of 3.3.1 (b) when 3.3.1 (b) is applicable.

4.3.7 Examination. Test samples and parts shall be carefully examined visually to determine conformance with all of the requirements of 3.3, except for 3.3.1. For 3.3.1 (a) and 3.3.1 (c) requirements the test samples and parts shall be examined both visually and by hand manipulation (as described in the two paragraphs) to determine that they meet all of the requirements of the two paragraphs (See 4.3.6 for compatibility test for 3.3.1 (b) requirements).

5. PREPARATION FOR DELIVERY

5.1 Cleaning. Test slabs and test buttons shall be flushed and/or immersed in fresh isopropyl alcohol, TT-I-735, Grade A, to clean them prior to packaging. Molded parts may be scrubbed if deemed necessary;

however, care must be exercised so that the surface texture of the parts remains undisturbed. Ultrasonic cleaning of parts is an acceptable alternate method.

5.2 <u>Packaging and Packing</u>. Test slabs, test buttons, and molded parts shall be packaged individually in heat-sealed polyethylene bags.

6. NOTES

- 6.1 <u>Intended Use</u>. The elastomeric material specified herein is intended primarily for use in aerospace sealing applications which require excellent resistance to solvent effects of hydrazine systems.
- 6.2 <u>Hydrazine is a hazardous chemical</u>. One not completely familiar with its use should refer to "Dangerous Properties of Industrial Materials" by N. Irving Sax.
- 6.3 Hydrazine Decomposition. Hydrazine decomposed by rubber generates gas pressure which can be measured by a gauge fitted to a closed storage tube. The amount of pressure rise is inversely related to the "compatibility index" of the rubber. Spurious results can be obtained if the rubber or apparatus is not cleaned thoroughly. In addition, the correct sample/propellant/ullage ratio must be used because a change in any one weight or volume can influence the pressure.
- 6.4 Non-Volatile Residue. A specific value or range can not at this time be specified for the amount of non-volatile residue from the propellant evaporation. Nevertheless, rubber formulation chemists can make evaluations of the quality of the rubber from these data.

APPENDIX E-2

MATERIAL SPECIFICATION (PRELIMINARY)

RUBBER

DESIGNATION: AF-E-124D

1. SCOPE

- 1.1 Scope. This material specification establishes the requirements for an elastomer compound intended specifically for seals.
 - 1.2 Classification. The designation of this material is AF-L-124D

2. APPLICABLE DOCUMENTS

SPECIFICATIONS

Federal

0-A-51

Acetone, Technical

TT-I-735

Isopropyl Alcohol

Other

MIL-P26536

Propellant, Hydrazine

MSC-PPD-2B

Propellant, Dinitrogen Tetroxide

OTHER PUBLICATIONS

American Society for Testing and Materials

ASTM D 15	Compound and Sample Preparation for Physical Testing of Rubber Products	
ASTM D 395	Compression Set of Vulcanized Rubber, Tests for	
ASTM D 412	Tension Testing of Vulcanized Rubber	
ASTM D 624	Tear Resistance of Vulcanized Rubber, Tests for	
ASTM D 1460	Change in Length of an Elastomeric Vulcanizate Resulting from Immersion	
•	in a Liquid, Test for	

American Society for Testing and Materials (Cont'd)

ASTM D 1708

Tensile Properties of Plastics by

Use of Microtensile Specimens,

Test for

ASTM D 2240

Indentation Hardness of Rubber

and Plastics by Means of a

Durometer, Test for

REQUIREMENTS

" / 'ASS. A.

3.1 <u>Material</u>. The material shall be AF-E-124D Elastomer, formulated and processed to meet the requirements of this specification. The composition of the base polymer shall be (by weight):

Approx. 60% Tetrafluoroethylene (TFE)

Approx. 40% Perfluoromethyl Vinyl Ether (PMVE)

< 2% Perfluorophenyl Vinyl Ether (PPVE)

As supplied by E. I. du Pont de Nemours & Company under the designation AF-E-124D.

The cure and post-cure schedule shall be as specified in Table I.

Table I. Cure & Post Cure Schedule

Press: 30 minutes/177°C (350°F)

Air Oven: Room Temperature to 150°C (300°F)
over 4 hours

24 hours/150°C (300°F)
24 hours/177°C (350°F)
24 hours/204°C (400°F)
Raised from 204°C (400°F) to
285°C (550°F) over 6 hours
24 hours/285°C (550°F)

All materials shall be of high quality and suitable for the purpose intended, and free of impurities reactive with nitrogen tetroxide, hydrazine, UDMH, MMH, or a 50/50 blend of hydrazine and UDMH.

- 3.2 <u>Physical Properties</u>. Physical properties shall conform to the requirements specified in Table I, for Oxidizer Service and Table II for Fuel Service. For Bipropellant Service the requirements of both Tables must be met.
- 3.3 Workmanship. Each molding shall be free of functional defects, foreign materials, and shall be uniform in quality; it shall also be free from mold flash unless otherwise permitted by the specific test method. There shall be no visible evidence of surface irregularities, such as unbonded sections, excessive holes or dents, blisters, and pits greater than 0.005 inch in height or depth. Small surface blemishes less than 0.005 inch in height or depth shall not be allowed when grouped closely together and cover more than 10 percent of the total surface area.
- 3.3.1 <u>Pigmentation</u>. Markings on the rubber surface which appear to be two dimensional (i.e., appear to be a discoloration rather than an inclusion) shall be acceptable as long as the following criteria are met:
 - (a) The discolored portion does not affect the flexed morphology of the rubber.
 - (b) The discolored portion does not react same compatibility requirement as in Tables I and II.
 - (c) The discolored portion does not show any tendency, upon repeated flexing, of separating from the rubber substrate.

4. QUALITY ASSURANCE PROVISIONS

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4.1 Qualification. Materials supplied to this specification shall have been qualified in accordance with all of the requirements of this specification.

Table I. Physical Properties for Oxidizer Service

Property	Requirement
Original:	
Tensile strength, psi, min	1500
Elongation, %	185 + 25
Hardness, Type A Durometer	78 - 90
Tear strength, lb/inch, min.	140
Following exposure to conditions as specified:	
Compression set, 22 hours at 200°F,	į
24-hour rest, % max. (Method B)	40
Fluid resistance, N_2O_4 immersions,	
100 hours at 160F, Air Dry at R.T. Days	
Tensile strength, % of original value (at 75°F) (min)	70
Elongation, % of original value (at 75°F)	85 - 150

Table II. Physical Characteristics for Fuel Service

Property	Requirement
Original:	
Tensile strength, psi, min	1500
Elongation, %	185 + 25
Hardness Type A durometer	78 - 90
Tear strength, 1b/inch, min.	150
Following exposure to conditions as specified:	
Compression set Method B, 22 hours at 70-75°F	15
22 hours at 200°F	40
24-hour rest, % maximum	
Fluid Resistance, N ₂ H ₄ immersion	1
100 hours at 160°F, Air Dry for 7 days at room	temp.
Tensile strength, (% of original value at 75°F)	90
Elongation, % of original value (at 75°F)	90
Compatibility, Pressure rise psi, max at 200°F (greater than control container)	15

- 4.1.1 Qualification Certification. The supplier submitting material for qualification shall provide certification that the materials submitted conform to all of the requirements of this specification and represent normal production run material.
- 4.1.2 <u>Qualification Sampling</u>. Qualification sampling shall include two test slabs prepared in accordance with the requirements of ASTM D 15 and two molded compression set buttons prepared in accordance with ASTM D 395, Type I.
 - CAUTION: No marking shall be affixed or applied to a sample, only to the package in which it is contained. (See Section 5 for cleaning, packaging, and marking requirements.)
 - 4.1.3 Qualification Testing. Qualification testing shall consist of demonstration of conformance to all of the requirements of this specification.
 - 4.1.4 Qualified Status and Requalification. A material that has been qualified in accordance with this specification require requalification for any change of raw materials and/or processing from that used for qualification.

4.2 Acceptance.

- 4.2.1 Acceptance Certification. The supplier of material shall certify that each shipment of material made to this specification conforms to all of the requirements of this specification. Certification shall include the actual results of laboratory tests to determine conformance.
 - 4.2.2 Acceptance Sampling.
- 4.2.2.1 Batch. A batch shall be the quantity of material compounded on a mill at one time.
- 4.2.2.2 <u>Preproduction Sampling</u>. Preproduction sampling shall include from each batch two test slabs prepared in accordance with the requirements of ASTM D 15 and two molded compression set buttons prepared in accordance with ASTM D 395, Type I.

- 4.2.2.3 <u>Production Sampling</u>. Production sampling shall include each production item.
 - CAUTION: No marking shall be affixed or applied to a sample or to a molded part, only to the package in which the item is contained. (See Section 5 for cleaning, packaging, and marking requirements)
 - 4.2.4 Acceptance Testing.
- 4.2.4.1 <u>Preproduction Testing</u>. Prior to the start of a production run for each batch of material the preproduction samples shall be tested to all of the requirements of this specification.
- 4.2.4.2 Production Testing. Each production item and subsequently the packaging for the item shall be inspected to determine conformance with the requirements of 3.3 and Section 5, respectively.
- 4.2.5 Rejection. Sample material and production items failing to meet the test requirements in 4.2.4 shall be rejected.
- 4.3 <u>Test Methods</u>. Qualification and acceptance testing of the material to the requirements of this specification shall be in accordance with the following:
- 4.3.1 Tensile Strength and Elongation. The tensile strength and ultimate elongation shall be determined in accordance with the requirements of ASTM D 412, using Die "D" for the specimens. Results shall conform to the requirements of Table I.
- 4.3.2 <u>Hardness</u>. The hardness shall be determined in accordance with the requirements of ASTM D 2240. Results shall conform to the requirements of Table I.
- 4.3.3 Tear Strength. The tear strength shall be determined in accordance with the requirements of ASTM D 624, using Die "B". Results shall conform to the requirements of Table I.

- 4.3.4 Compression Set. The compression set shall be determined in accordance with the requirements of ASTM D 395, Method B at the aging time and temperature specified in Table I. Results shall conform to the requirements of Table I.
- 4.3.5 <u>Fluid Resistance</u>. The procedure outlined below is used to determine the fluid resistance and the relative compatibility of the rubber specimens.
 - (a) Cut three specimens of the rubber from a test slab, using the ASTM D 1708 die.
 - (b) Clean the specimens using in sequence a detergent solution. O-A-51 acetone, and finally reagent grade isopropyl alcohol. Dry for 5 minutes at ambient temperature in a dirt/dust-free environment. Do not handle with bare hands.
 - (c) Measure the *length and weight of each specimen.
 - (d) Using rubber gloves which have been pre-rinsed in isopropyl alcohol and dried, place the rubber specimens into the bottom of an 85 cc glass Acrosil Compatibility Tube (e.g. VWR Scientific Company Catalog No. 62810-000) precleaned and dried as in (b) above. The tube must have at least a 100 psig safety rating. The primary cap seal must be Teflon and the secondary seal shall be cut from the test slab.
 - (e) Add slowly 50 ml of MIL-P-26536 hydrazine to each tub .
 - WARNING: This step must be performed with care by a propellant handler; hydrazine is a dangerous chemical.
 - (f) The Aerosil Compatibility Tube shall be fit_ed with all 304 CRES fixtures, tubing, gauges, and valves as follows:

To the threaded flange cap attach a "T" fitting. From one leg of the "T" attach a 1/8 inch tubing (See (g) below) leading to a compound Bourdon-type pressure gauge (calibrated in inches of Hg on the vacuum side and in psia on the pressure side; a convenient size is 0 to 100 psia).

CAUTION: Do not use bulkhead fittings to interface from pipe threads to other types such as AN, because bulkhead fittings require 0-rings for sealing. Fitting threads shall be wrapped with TFE pipe tape prior to assembly. The whole assembly, prior to fitting on the tube, shall be rinsed in isopropyl alcohol and dried (See (b) above).

- (g) Place the Aerosil Compatibility Tube and the valve inside an oven at room temperature. The oven shall have been precalibrated to the desired temperature (See Table I). Pass the 1/8 inch tubing (see (f) above) through the over wall so that the pressure gauge can be maintained near ambient temperature. The total volume of the 85 cc tube, fittings, gauge, valve, and tubing should be approximately 95 cc.
- (h) Place the oven in a bunker or test cell which has a viewing window through which the pressure gauge may be observed.
 - <u>WARNING</u>: A power switch for the oven shall be placed outside the test bay in order to turn off the oven if the pressure approaches the safe limit of the glass compatibility tube. If this occurs, the oven must be turned off immediately inasmuch as excessive pressure can be quite dangerous.
- (i) Connect the valve to a vacuum pump isolated from the hydrazine by a cryogenic trap (CRES or glass tubing and fittings from the valve to the trap) and evacuate the apparatus for several minutes, or until the hydrazine ceases to bubble (outgassing has ceased). The compound gauge must indicate a vacuum of at least 28 inches of Hg. Close the valve and disconnect the vacuum pump.

- (j) Check the system for zero leakage by reading the compound gauge after 30 minutes. Eliminate any leakage. When the system is leak-free, turn on the oven to the calibrated temperature and when the oven reaches the desired temperature, start the storage time.
- (k) At the end of the required storage time (or before, see (h) above) record the pressure and turn off the oven. Cool to ambient temperature (at least several hours), record the pressure again and vent the valve to relieve any residual pressure.
- (1) In a well-vented fume hood remove the flange cap, decant the propellant and, while still working in the fume hood, place the specimens on laboratory wiping tissues (Kimwipes, Kimberly-Clark Company, are satisfactory) and carefully blot dry. Then remove the specimens, using the same clean gloves described in (d) above.

<u>WARNING</u>: Do not use a sulfite-type paper for blotting inasmuch as this paper in contact with hydrazine is a fire hazard.

- (m) Measure the blotted samples for length and weight. Calculate the length and weight change. The volume change may be calculated from the length change in accordance with ASTM D 1460.
- (n) In a clean, pre-weighed round bottom flask carefully evaporate to dryness an aliquot of the decanted propellant and then calculate the propellant's non-volatile residue in milligrams per 100 milliliters (mg/100 ml) as the units.

WARNING: This step must be done carefully and in a safe place by

a propellant handler; the procedure can be very dangerous.

(o) A control propellant sample shall be processed in accordance with Steps (d) through (n) exactly as above except that the rubber specimens shall not be introduced.

- (p) The non-volatile residue from Step (o) shall be compared with that from Step (m) (with the rubber specimens). See 6.5.
- (q) Allow the exposed rubber specimens (from Step (m) above) to air-dry for 24 hours and then measure and test them for the properties specified in Table I. The results shall conform to the requirements of Table I.

The results of this test shall conform to the requirements of Table I and also the compatibility portion shall conform to the requirements of 3.3.1 (b) when 3.3.1 (b) is applicable.

4.3.7 Examination. Test samples and parts shall be carefully examined visually to determine conformance with all of the requirements of 3.3, except for 3.3.1. For 3.3.1 (a) and 3.3.1 (c) requirements the test samples and parts shall be examined both visually and by hand manipulation (as described in the two paragraphs) to determine that they meet all of the requirements of the two paragraphs (See 4.3.6 for compatibility test for 3.3.1 (b) requirements).

5. PREPARATION FOR DELIVERY

- 5.1 Cleaning. Test slabs and test buttons shall be flushed and/or immersed in fresh isopropyl alcohol, TT-I-735, Grade A, to clean them prior to packaging. Molded parts may be scrubbed if deemed necessary; however, care must be exercised so that the surface texture of the parts remains undisturbed. Ultrasonic cleaning of parts is an acceptable alternate method.
- 5.2 Packaging and Packing. Test slabs, test buttons, and molded parts shall be packaged individually in heat-sealed polyethylene bags.

6. NOTES

6.1 <u>Intended Use</u>. The material specified herein is intended for Aerospace Seal Applications requiring a high degree of compatibility with nitrogen tetroxide and hydrazine base fuels.

- 6.2 <u>Nitrogen Tetroxide Test Techniques</u>. Nitrogen tetroxide is a hazardous and toxic chemical. Testing and handling must be accomplished only by experienced personnel. Specific test techniques should be established and documented for the required compatibility tests.
- 6.3 <u>Hydrazine is a hazardous chemical</u>. One not completely familiar with its use should refer to "Dangerous Properties of Industrial Materials" by N. Irving Sax.
- 6.4 Hydrazine Decomposition. Hydrazine decomposed by rubber generates gas pressure which can be measured by a gauge fitted to a closed storage tube. The amount of pressure rise is inversely related to the "compatibility index" of the rubber. Spurious results can be obtained if the rubber or apparatus is not cleaned thoroughly. In addition, the correct sample/propellant/ullage ratio must be used because a change in any one weight or volume can influence the pressure.

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6.5 <u>Non-Volatile Residue</u>. A specific value or range can not at this time be specified for the amount of non-volatile residue from the propellant evaporation. Nevertheless, rubber formulation chemists can make evaluations of the quality of the rubber from these data.